THE EFFECT OF FERTILISATION ON THE ANATOMICAL AND CHEMICAL CHARACTERISTICS OF *EUCALYPTUS GLOBULUS* AND THEIR INFLUENCE ON THE RESULTANT PULP AND PAPER PROPERTIES

by

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STATEMENT OF ORIGINALITY

Except where specific acknowledgement is given, this thesis is my own original work.

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ABSTRACT

UMORU, B. (1977) The effect of fertilisation on the anatomical and chemical characteristics of *Eucalyptus globulus* and their influence on the resultant pulp and paper properties.

Fertilisation did not significantly reduce fibre length or basic density. The decrease in basic density is more than compensated by the significant increase in the volume of wood produced. High levels of fertilisation significantly reduce double wall thickness and increase lumen diameter, thus increasing collapsibility levels in fibres and consequently produce pulps of high strengths. There was a significant increase in the amount of methanol-soluble extractives in the wood at the higest level of fertilisation but a lower lignin content. The gradual dwindling in effect of fertilisers was observed in most of the anatomical characteristics measured. Consequently it is suggested that fertiliser application should be spread over a few years. On the whole, fertilisation had no deleterious effects on anatomical properties considered most important in the pulp and paper industry.

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CHAPTER 1

INTENSIVE EUCALYPT PLANTATION FORESTRY - AN ANSWER TO INCREASING WOOD FIBRE DEMAND FOR THE PULP AND PAPER INDUSTRY

1.1 Introduction

At present the pulp and paper industry is mainly dependent for its fibrous raw material on forest trees although bamboo and bagasse may also be used. In the past the industry has been rather complacent about alternative raw material for pulp and paper because of its reliance on extensive utilisation of temperate native forests, especially conifers, which in many parts of the world have carried, and still carry in some, abundant supplies of timber suitable for conversion to all kinds of pulp and paper products. Hardwood species were generally not well regarded. This position however has changed since, in many parts of the world, native forests have either been denuded or are insufficient to meet the growing demands of the industry. Efforts are now being made to establish plantations of selected species for industrial utilisation, including the pulp and paper industry. See Tables 1.1 (a) and (b).

In Australia, the relative abundance of old growth forests and the scattered location of the pulp and paper industry gave rise to extensive forest management with little or no incentive for increased fibre productivity. In recent years, however, there has been a shift of emphasis from extensive management of natural forests to intensive plantation forestry everywhere, a shift brought about by many factors,

TABLE 1.1 (a)

Estimated additional roundwood requirements for the years 1975 and 2000 in developing countries for paper and paper board, wood used in the round, fibreboard and particleboard. Also shown are plantation areas, rate of planting and yield expectations.*

Region	Estimated requirements for 1975 (a) Million cubic metres				Area of existing plantations (1964) 1000 hectares			Estimated Annual Yield as of 1975 Million M^3	Rate of Planting (1964) Million ha/yr	Estimated additional requirements[a] for year 2000 for paper & paperboard[b] Million M^3	Estimated annual yield as of year 2000 if current rate of plantation establishment is maintained Million M^3
	Paper & Paperboard	Wood used in the round	Fibroboard & Particle-board	Total	Conifers	B/leaved	Total				
Latin America	11.3	2.1	1.5	14.9	500	1100	1600	16.00	0.150	70	53
Near East	0.7	1.0	0.2	1.9	150	1500	1650	1.50	0.015	5	5
Asia-Pacific	7.2	4.7	0.5	12.4	15	135	150	16.50	0.120	45	46
Africa	2.4	3.8	0.3	6.5	240	735	975	9.75	0.050	20	22
Total	21.6	11.6	2.5	35.7	905	3470	4375	43.75	0.335	140	126

* Source: F.A.O. Staff, Unasylva 1965, No. 79.

a Over 1959-1961 average annual consumption.

b Estimates confined to Paper and Paperboard, only product for which meaningful predictions can be made this far into the future.

Estimates assume average rates of growth of 2.5% per annum in population and 2% per year in per caput income for each region. They also assume an average requirement of 2.75 cubic metres of roundwood per ton of paper and paperboard in each region.

Table 1.1 (b)

Comparison of areas of man-made forests in 1965 and those planned for 1985*

Region	1985 (million hectares)	1965	Ratio 1985/1965
Africa	4.1	1.6	2.6
Asia	8.5	3.5	2.4
Australasia	1.8	0.8	2.3
Europe	13.0	6.7	1.9
Latin America	4.5	0.8	5.6
Near East	0.16	0.05	3.2
North America	27.8	10.6	2.6
TOTAL	59.86	24.05	Average 2.5[a]

* Source: World Symposium on Man-Made Forests and Their Industrial Importance. Canberra April 1967. Figures are not complete since some countries declined to make forecasts.

a It can be seen for reporting countries that present plans are to have 2½ times the area planted in 1985 as in 1965.

among them:

(a) The shrinkage land base available to industries. The present public pressure to have more areas of scientific interest and natural beauty conserved as parks or recreation areas and removed from productive use is unlikely to decrease.

(b) Land has been taken up by increased urban development and roads.

(c) The increasing purchase price of land and increasing rates and taxes.

(d) Wood processing plants are becoming much bigger and strong incentives to increase fibre productivity have developed.

It has become essential for forestry and wood-based industries to use land as efficiently as possible. This has led to the use of fertilizers to improve growth on poor sites and fertile sites; fertilization has helped to reduce rotation age. In evaluating the results of fertilization it is important to know if it causes important changes in the chemical composition and the physical properties of wood.

In recent years, many investigations relating to response of forest trees to fertilization have been carried out, especially the application of nitrogen. A comprehensive bibliography has been compiled by White and Leaf (1957).

In addition to increases in yield one of the major benefits of intensive forestry in general is the ability to produce relatively uniform raw material arranged in a methodical way. This permits mechanized operations and reduces labour requirements.

There is a growing interest in the harvesting of very young trees and utilizing a higher proportion of the fibre from a site. This is evident in the increasing proportion of young plantation grown material included in pulpwood furnishes to many mills (Cromer 1971). More information on the wood characteristics and utilisation of fast grown material is essential, especially in those species for which such information is available only with respect to relatively slowly grown indigenous trees. Many Australian eucalypts are in this category. The next section deals with the forest resources of the world and the role eucalypts as a genus plays or can play especially as a source of fibre for the paper industry.

1.2 Forest resources - general world trends and prospects

The production of wood, in all its utilisable forms, is still the most important part of forestry despite the emphasis on conservation and preservation of forest resources in some of the more developed parts of the world. A vast range of other forest products such as gums, resins, tannins, latex, food for many living creatures and so on is also produced.

Most of the data used in this section are based on F.A.O. forecasts because of the difficulties in getting accurate actual data or lack of any kind of data in some parts of the world.

Of the 1000 million m^3 of industrial wood consumed throughout the world in 1961, the majority was in saw logs, three times as much as wood for pulp and more than ten times as much as the amount of wood used for panels of various kinds.

This pattern has since changed. It was estimated that by 1975 (F.A.O. 1963) the world would require about 560 million cubic meters more wood annually than it did in 1961. About 450 million cubic meters of this additional quantity would be required for industrial use - a rise of 43% in 14 years. A growing share of industrial wood, will be required for pulp products and wood based panels; consumption of which was estimated to rise by 110% and 150% respectively over the period. This compares with a 23% rise in sawn wood consumption and virtually no change in the figure for round wood consumed as such.

This shift in the pattern of demand means that different dimensions, different quantities and different wood properties have become more important. Three-fifths of this increase in industrial wood volume was estimated to be required for chipping or pulping between 1961-1975. Below is the estimated growing stock of world forest resources.

Table 1.2

Growing stock in I,000 million m^3 including bark (F.A.O. 1963)

Region	Coverage* %	Total	Conifers	Non-Conifers
North America	62	44	33	11
Central America	14	0.8	0.2	0.6
South America	51	78	0.5	77
Africa	12	3.8	0.1	3.7
Europe	98	12	7.6	4.3
U.S.S.R.	100	79	66	13
Asia	45	17	6	11
Pacific Area	70	3.8	0.4	3.4
WORLD	55	238	114	124

* Percentage of forest land (or of forest where applicable) on which data are reported.

Table 1.3

Expected change in the world's industrial wood balance 1961-1975 (million cubic meters of round wood and wood raw material equivalent)*

Region	Consumption 1961	Consumption 1975	Production 1961	Production 1975	Surplus (+) or deficit (-) 1961	Surplus (+) or deficit (-) 1975
Europe	259.4	376	224.3	272	-21.1	-79
U.S.S.R.	243.2	305	256.8		+15.8	
Canada	32.5	44	89.0		+56.5	
U.S.A.	288.0	376	250.3	326	-37.7	-50
Total North America	320.5	420	339.3		+18.8	
Latin America	39.9	76	38.5		-1.4	
Africa	25.0	36	25.6		+0.6	
Near East	9.5	14	6.4		-3.1	
Far East[a]	41.5	68	45.3		+3.8	
Japan	63.0	112	48.4	72	-10.4	-30
Mainland China	34.0	62	34.0			
Pacific	18.1	26	15.4	21	-2.0	
World	1054.1	1490	1034.0		+1.0	

* Source: Wood World Trends and Prospects, F.A.O. Unasylva, Number 80-81, 1966.

a Excluding Japan and Mainland China.

Consumption of industrial wood is concentrated in the technologically advanced countries of the world. These countries accounted for 70% of the increase between 1961 and 1975. In some of the leading wood consuming countries, domestic wood supply is no longer keeping pace with expanding demand.

A rapidly increasing supply deficit is forseen for Europe as a whole, except, perhaps for Scandinavia. Net imports were expected to supply 21% of industrial wood requirements in 1975 compared with 8% in 1961.

Japan has also emerged as a major wood deficit area and is buying large quantities of wood chips from Australia and elsewhere, large volumes of tropical hardwood saw and veneer logs from the South-East Asian region. The United States of America will also import increasing quantities of wood or wood based products. The additional import requirement of other parts of the world are likely to be comparatively small, though their aggregate requirements are also rising as a result of educational programs e.g. Nigeria and Somalia. Nigeria has in effect become a net importer of wood, despite her extensive forests because of the increasing amount and cost of imported paper and paper products as against her wood exports.

In many countries rapid growth in the market is permitting a greater degree of domestic processing, whilst man-made forests are beginning to make a major contribution to wood supply in some areas previously suffering a wood shortage.

To meet increasing needs, it is expected there will be greatly increased exports of sawn soft wood, from Canada and U.S.S.R.; pulp and paper mainly from North America and Northern Europe; and veneer quality hardwoods, mainly from the tropics.

The continuing increase in the world's need for pulp and paper products has induced research groups and individuals to study methods which might improve utilisation of the raw materials for paper and paper products. Recycling of waste paper has been on the increase. In recent years both recovery and utilisation of waste paper have tended to rise in developed countries and it is widely expected that this trend will continue in future. See Table 1.4. It has been estimated that in 1985 U.S.A. will be recycling 26% of their total pulp consumption (OECD 1976).

An increase in the supply of fiber for industry can be achieved in three ways (Pēterī 1952):

(1) improving existing processes or developing new ones to increase the yield of pulp for paper making;

(2) improving the paper making qualities, and strength characteristics of pulps so that the basis weight of paper for a particular product may be reduced thus effectively increasing supply;

(3) the use of other fibrous materials and recycling of waste paper.

Paper is made from bamboo in India and in the Phillipines from sisal hemp. Many other processes are being developed,

Table 1.4

Recovery and utilisation of waste paper for some countries*

Countries	1965 (%) Recovery Rate[a]	1965 (%) Utilisation Rate[b]	1974 (%) Recovery rate	1974 (%) Utilisation Rate
Canada	14.7	4.2	18.0	6.4
U.S.A.	21.7	23.0	22.3	20.6
Japan	37.4	35.3	39.2	37.1
Norway	20.0	7.1	20.5	7.4
Sweden	21.1	5.9	28.0	7.3
	(25)[1]		(26)[1]	
Finland	19.7	4.1	17.2	3.2

* Source: OECD 1976

a Recovery rate is the ratio of waste paper collected to paper and paper board apparent consumption.

b Utilisation rate is the ratio of waste paper consumption to total consumption of fibrous materials in the manufacture of paper and paper board.

1 Adjustments have been made in the recovery rate of Finland since substantial quantities of paper and paper board are converted into products which are then exported, thus figures of apparent domestic consumption may substantially overstate actual domestic consumption of paper and paper board and therefore understate the recovery rate.

tropical hardwoods being one of the raw material sources that could help to alleviate shortages of raw materials in the industry.

There is now much less bias against hardwoods as a source of fibre for the paper industry. Australia has used eucalypts very successfully; so has Portugal and Spain where introduced eucalypt species are the main source of raw materials for the pulp and paper industries.

1.3 Role of Eucalypts in the paper industry and timber market

The potential of eucalypts as plantation species has been recognised throughout the world. Eucalypts have been established in plantations in many parts of the world where growth conditions are suitable. They have been cultivated for more than a century in Spain and Portugal where high standard silvicultural and management practices have been reached, albeit with relatively labour intensive methods (Cromer 1975). See Table 1.5 for areal distribution of eucalypts. Today there are a number of large mills entirely dependent upon plantation eucalypts for short fibred pulp; the major species in Portugal and Spain being *Eucalyptus globulus*. The establishment in Australia, Spain and Portugal and elsewhere of pulp and paper industries using eucalypts as raw material has also revealed a number of problems.

Table 1.5

Area distribution of Eucalypt species as compared to Conifers and other broadleaved species (1965) in plantation*

Region	Conifers	Eucalypts	Other broad-leaved species
	(1000 hectares)		
Africa[1]	667.3	454.8	474.9
Asia	620.8	93.7	2,752.0
Australasia	723.3	18.8	25.2
Europe excl. U.S.S.R.	4,913.1	148.7	910.1
Latin America	405.9	249.8	172.5
Near East	4.0	10.3	31.6
North America	9,819.0	-	829.0

* Source: World Symposium on Man-made Forests and their Industrial Importance. Canberra April 1967.

1 Eucalypts alone are almost equal in distribution to all other broad leaved species in plantations.

Currently, Australia produces more than 231,000 tons of pulp (excluding ground wood) and about 90,000 tons of newsprint from eucalypts. Forty years ago, the wood of mature eucalypts was considered unsuitable for use as raw material for pulp (Hillis, 1975). With the success of this industry, the advantages of hardwood pulps have become more evident. Eucalypts pulps are no longer regarded as inferior or suitable only as fillers. Rydholm has said that the best furnish for fine papers is a mixture of Eucalypt and softwood (Hillis, Lecture, 1975).

Eucalypts are suitable as plantation species because some of them grow rapidly and are therefore preferred for short rotation crops. Harvesting ages of 5-10 years are common in Africa (Myburgh, 1967), South America (Lean, Borges, 1967) and other countries. Although the high incidence of growth stresses in such trees could hinder their utilisation as construction and building material, this defect does not affect their use for reconstituted wood products. However, brittle heart in some trees containing a high percentage of broken fibres can reduce their value for pulp (Hillis, 1975).

The eucalypts thrive in warm temperate and tropical areas especially where there is periodic or seasonal drought. Because of this, eucalypts are being planted in arid areas where indigenous species often do not do well. Thus eucalypts have been used in Nigeria for reclamation of mined areas.

The marked response by eucalypts to fertilizer application and cultivation is well documented. Growth rates in the driest regions of Portugal range from 4-10 cubic meters per hectare per year and in the more favourable regions, rates between 15 and 30 cubic meters per hectare per year are obtained (Cromer, 1975). These figures represent over-bark volume of wood to a small-end diameter limit of 8cm, over a 10 year rotation.

In the long term in some countries, the ability of their forests to meet their domestic demand rests on the exotic eucalypts. In the Northern dry areas of Nigeria for example more intensive forestry operations can be carried out mechanically.

Eucalypts can of course still be used in more traditional ways such as for fuelwood, sawn wood, poles, fence posts, shelter belt plantings and for fruit cases and veneer. In Iraq, eucalypt wood is intended to be used for making rayon (F.A.O. 1975). A cottage industry has developed around the extraction of essential oils from the leaves of *Eucalyptus globulus* in India grown under a rotation of 5 years.

1.4 Purpose of this study

A.P.M. Forests began experiments in eucalypt plantation establishment in 1953 (Alexander, 1959), using blood and bone fertiliser at time of planting. Since then many other experiments have indicated that plantation grown eucalypts respond well to fertiliser application (Hall and Richmond, 1961, Cromer, 1971).

In 1969 another experiment was set up using 4 levels of fertiliser application with 4 replications. The fertiliser used was a commercial blend of ammonium phosphate and ammonium sulphate (HyGold 18) containing 18% N and 8% P.

Following the complete tree utilisation concept proposed by Young (1964) and very short rotation "silage" forestry expounded by McAlpine et al (1966), biomass, nutrient uptake and pulping evaluation studies were included in the experiment. The experimental design is shown in Table 3.1.

In this study, deriving material from the same experiment, attention was concentrated on the effect of the fertiliser application on the anatomy of the wood and how this in turn affected the properties of the paper derived from the wood. The effect of fertilisation on the chemical nature of the wood and bark was also investigated.

Evaluation of the pulp and paper properties was carried out in A.P.M. Research Laboratory at Fairfield, Victoria. Unless otherwise specified, APPITA standards have been used in the evaluation.

CHAPTER 2

DENSITY--LITERATURE REVIEW

2.1 Introduction

Density can be defined as the weight (in gms or pounds) of a measured volume (in cubic centimeters or cubic feet) of substance. In wood this varies (a) according to the amount of wood substance (cell wall material) present per unit volume, (b) according to the amount of infiltration in the wood and (c) according to its moisture content. Strictly, actual wood density is concerned with the amount of all wall substance per unit volume. Thus figures of relative density include both (a) and (b) since it is impracticable to separate the infiltration products from the wood substance proper. In the literature and in wood technology the term specific gravity (ratio of weight of the wood substance to the weight of an equal volume of water) is often used.

Due to the heterogeneous nature of wood and the fact that wood volume changes in the presence of moisture, the measurement of wood density is complicated. Thus at least four different kinds of measure of wood density can be made:

(1) Basic Density: oven dry mass/green or swollen volume in gms/cm^3 or kg/m^3. This is a derived unit and not measureable directly but because it is easily reproduceable and eliminates other variabilities it is commonly used.

(II) Air density: air dry mass/air dry volume of wood in gms/cm^3 or kg/m^3. It varies according to the ambient temperature and relative humidity. It also varies according to how the wood has reached a particular moisture content either by desorption from higher moisture content or adsorption from lower moisture content. Thus this has been standardised in different countries e.g. Australia uses 12% moisture content, and for tropical countries 15% moisture content at which air dry density is expressed.

(III) Green density: mass of the green volume of wood/ swollen volume in gms/cm^3 or kg/m^3.

(IV) Oven dry weight/volume of wood at a specified moisture content still in the same units.

In this study, wood density means basic density where it is not specified.

Of all the wood properties basic density has come to be regarded as an important indicator of some of the properties of wood because it is also easily reproduceable. It may be used as index for many strength values and for working qualities in solid wood. Some wood strength properties, like stiffness vary almost directly with basic density while for others the relation is less direct e.g. toughness varies almost as the square of density. Dadswell and Nicholls (1959) found that in *Pinus elliotii* var. *elliotii*, basic density was a good index of average cell wall thickness.

Green density is important in problems of handling and transportation. It can be used to estimate the pulp yield. Basic density may be helpful in predicting certain pulping and paper characteristics such as conformability, sheet density and most importantly paper strength properties such as tear, burst and crushing strengths. Consequently studies relating to basic density and its variations, have been of increasing economic and technical interest especially in those countries concerned largely with hardwood pulping or with the export or import of hardwood chips.

The purpose of this section of the study was to investigate the effect of fertilisation on the basic density of *Eucalyptus globulus*, the variation of basic density with height and its effect on the proprerties of pulp and paper derived from it. The pulp and paper was made at the research laboratory of A.P.M. Pty. Limited, Fairfield, Victoria.

2.2 Basic density variations

There are contradictory early reports on the changes in the basic density in different trees. Some reported a decrease in basic density with increasing height for *Betula* species (Stauffer, 1892), for *Pinus strobus*, *Tsuga heterophylla*, *Acer saccharum* and *Quercus alba* (Myer, 1930) for *Pinus sylvestris* (Schwappack, 1897), and for *Pseudotsuga menziesii* (Sterns, 1918). Others reported an increase with increasing height for *Abies alba* (Bertog, 1895) and for *Picea glauca* (Hale and Fenson, 1931). Eichorn (1895) reported no predictable variation with increasing height for basic density of *Quercus* species.

Stauffer (1892) also reported an increase in basic density with increasing age for *Betula* species while Eichorn

(1895) reported a decrease in *Quercus* species.

Chevandier and Wertheim (1849), Schwappack (1897) and Sterns (1918) reported a radial increase of basic density outwards from the pith in several soft and hardwoods.

Recent research especially in softwoods shows in the main that density increases with the age of the tree and decreases with increasing height (Spur and Hsiung 1954), Wellwood (1960), Schmidt and Smith, (1961).

However Wellwood and Jurazs (1968) working with 73 trees of *Thuja plicata* observed that basic density decreased from pith to bark and increased with increasing height. Such apparently contradictory evidence has also been reported in some softwood species by Wahlgren *et al*, (1966). Tackle (1962) and Conway and Minor (1961) working with *Pinus contorta* and *P. ponderosa* respectively found that density varied little with height particularly above the butt (1 meter).

Baker and Shotaffer, (1968) found that basic density variation with height in 25-year old *Pinus resinosa* differ between crown classes. In the dominant and sub-dominant trees wood density decreased with height in the lower bole and increased at higher levels but in the suppressed trees wood density increased consistently with height.

In some hardwoods especially ring porous species basic density commonly decreased from the butt to the central bole, then increased within the crown, e.g. in *Quercus falcata* (Hamilton, 1961), *Q. Sessiflora* (Todoravski, 1961), *Swistenia macrophylla* (Briscoe *et al*, 1963) and *Eucalyptus grandis* (Bamber *et al*, 1963). Density increased with height in *Populus* species (Gotze, 1964) and in the lower half of the bole of *Eucalyptus regnans* (Dargavel, 1968). On the other hand little predictable variation in density at different heights was observed in Japanese giant poplar *Populus japono-gigas* (Inokuma *et al*, 1956) and *Liquidamber styraciflua* (Carpenter and Hopkins, 1966).

Taylor (1973) working with 15-year old *E. grandis* (Hill) maiden grown in South Africa found the specific gravity decreased sharply with hieght from 1.5 to 4.6 metres and then followed by a consistent increase. He also found an initial increase in specific gravity at breast height followed by a pronounced increase in the specific gravity of outer sections. At 10.7 metres, the distance from pith had a less pronounced effect on specific gravity and at 22.9 metre level, there was almost a linear decrease in specific gravity as distance from the pith increased. The same trend was found in Zambian grown *E. grandis* (Hans, and Burley, 1972). They observed a decrease in basic density with height up to 15% of tree height and an increase at 35% height level.

Higgs (1969) and Davidson (1972) reported a complex pattern of basic density variation with respect to height and distance from the pith in *E. regnans* and *E. deglupta* Blume respectively.

Nicholls and Phillips (1972) found no clearly defined pattern of basic density variation either with position in the tree or age in coppice and seed propogated trees of *E. virminalis*.

2.3 The effect of fertilisation on wood density

Many studies have shown that fertilising forest trees increased growth rates and that such practices might also modify wood properties.

The effect of fertilisation on wood properties has been discussed by Possey (1964), Bonneau (1966), Boyd (1967), Fielding (1967), Karth (1967) and Klemm (1968). The literature refers mainly to softwoods and suggests that mineral fertilisers usually caused decreases in the average fibre length and average basic density. The few hardwoods that have been investigated showed the same response (Boyd, 1967).

Klemm (1968) reviewing this topic felt the response to fertilisation was affected by the nature of the stand in relation to prevailing climatic and edaphic conditions; whether the stand was on a fertile site, if periodic droughts occurred in the area. Paul and Marts (1954) found that irrigation of fertilised trees of longleaf pine (*Pinus palustris*) caused a remarkable increase in the percentage of summerwood as compared to the percentage of summerwood in trees where fertilisation was in dry years.

Zobel *et al* (1961) examining the effects of 3 heavy applications of nitrogen (179 kg/ha), phosphorus (90 kg/ha) and potassium (90 kg/ha) fertiliser for 3 consecutive years on the wood properties of 16-year old *Pinus taeda* found the basic density of the wood produced seven years after fertilisation was considerably less than that produced during the preceeding seven years.

Seibt (1963) found a decrease of up to 2-7% in the basic density of *Pinus sylvestris*, *Larix leptolepis* and *Picea abies*, following fertilisation with a mixture of calcium, nitrogen, phosphorus and potassium at time of planting. In a similar study using 30-year old *Pseudotsuga menziesii* Erickson and Lambert (1958), observed a decrease of about 8% over the 4 years following fertilisation using the same fertiliser mixture.

Williams and Hamilton (1961) found the basic density of 8-years old *Pinus elliotfii* fertilised with nitrogen alone, nitrogen plus phosphorus and phosphorus only, was less than the density of the control two years later. The greatest decrease was found with nitrogen application only, then nitrogen and phosphorus and phosphorus only respectively. Sastry (1967) also observed a similar decrease in density of 20-23-years old *Pseudotsuga menziesii* treated with nitrogen, phosphorus and potassium, although the effects were not satisfactorily significant due to the smallness of the sample. Siebt *et al* (1968) also reported that fertilisation gave a decrease of average basic density associated with an increase in the production of early wood.

In hardwoods Bosdorf (1968) working with 4 varieties of *Populus* found that density, two years after fertilisation with different combinations of nitrogen, phosphorus and potassium, was less than before treatment. Jakimov (1966) also reported a decrease in basic density in wood from poplar plantations in Russia, following fertilisation. Higgs (1969) also found that an application of mixed fertiliser of nitrogen ($(NH_4)_2 SO_4$ - 180 kg/ha) phosphorus (ca super-phosphate 22% of P - 90 kg/ha) and potassium ($K_2 SO_4$ - 90 kg/ha) to 28-years old *Eucalyptus regnans* (broadcast manually) resulted in the formation of wood with a significantly lower basic density. More recently Hans *et al* (1972) reported a non-significant decrease in basic density of four-year old *Eucalyptus grandis* grown in Zambia following fertilisation with three levels of NPK and boron.

Taylor (1973) observed no significant difference in the specific gravity of *E. grandis* grown in South Africa due to different growth rates.

CHAPTER 3

DENSITY INVESTIGATION ON SIXTEEN TREES OF *EUCALYPTUS GLOBULUS*

3.1 Introduction

Four 6-year old trees, were selected from each of four levels of fertiliser application with Hy-Gold 18. Details of the applications are shown in Table 3.1. A tree was also selected from treatment C, whose diameter at breast height was closest to the mean diameter of Plot C. for height sampling.

The 16 trees were felled and discs cut out at breast height. From the tree selected for height sampling discs were cut at 0.25m , 1m, 3m, 5m and 7m above ground level.

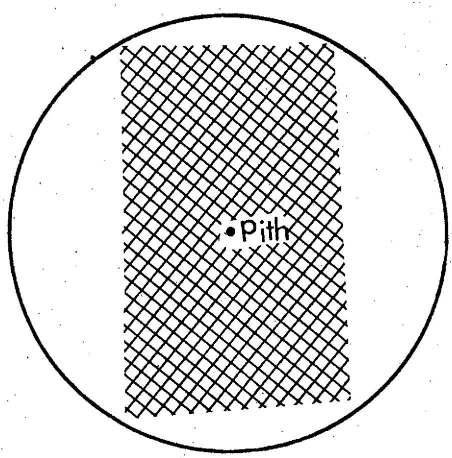

Fig 3.1 (a) Normal stem

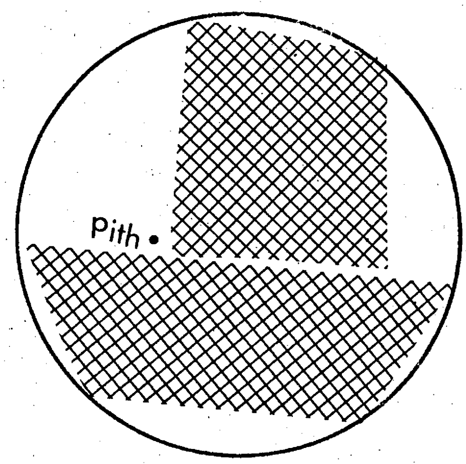

Fig 3.1 (b) Eccentric stem

Radical strips were cut from the discs as shown in Fig 1 (a) and from the strips irregular sample blocks were removed. In discs with eccentric growth, two samples were taken as shown in Fig 1 (b). The small samples were cut in such a way that all or most growth rings were represented. Since the trees were only 6-years old, it was not difficult to do this. This break-down was done in the workshop, using a small saw.

The sample blocks with rough surfaces due to crushed fibres were smoothed with a pocket knife. This was necessary to ensure that no error was introduced during weighing through loss of fibres.

3.2 Determination of density

Details of various gravimetric methods have been given by Smith (1954, 1955), and Phillips (1965) and one of these, based on immersion in water was used. It involves determining basic density by direct measurement of the oven dry weight and the saturated or green volume.

The saturated volume was determined using the water displacement method for irregularly shaped wood blocks of Heinrichs and Lassen (1970). The balance used was an adapted Mettler P-5 which allowed very rapid weighings to be made.

The blocks were autoclaved for an hour at 60°C and 109.24 kilo pascals pressure to remove some air and thus facilitate saturation with water. The steaming also helps to reduce the possibility of cell collapse in the sample blocks.

The blocks were then weighted down in a trough of water and left for 10 days after which they sank without weights. To ensure complete saturation with water, the blocks were subjected to half an hour of vacuum followed by pressure

Table 3.1

Quantity of fertiliser applied in the first 2 years.*

Quantity & time applied (kg/ha)	Treatment			
	A	B	C	D
HyGold 18 (18%N and 8%P)				
At planting (spot, 85g/tree)	0	188	188	188
At 8 months (strip, 170g/tree)	0	0	377	377
At 15 months (broadcast)	0	0	0	565
Total:	0	188	565	1130
Nitrogen (total)	0	34	101	202
Phosphorus (total)	0	15	45	90

* Data provided by Australian Paper Manufacturers.

of about 1043.5 KPa.

The blocks were then weighed using the Mettler P-5 after wiping the excess water from their surfaces with a cloth. The blocks were weighed in water in an open sided container suspended from a hook underneath the balance and attached to its weighing mechanism. Care was taken to ensure that the container did not touch the sides of the big container filled with water. See Fig. 3.2 (a) and (b).

The difference between the weight in air and the weight in water gave the volume of each block in cubic centimetres.

The blocks were then dried under vacuum, first at about 60°C for two days to prevent collapse and subsequently at 100°C for another 6 days until no further loss of weight was registered. This was checked by test weighing 3 sample blocks until they maintained a constant weight.

The blocks were quickly transferred from the vacuum drier to a desiccator, 4 blocks at a time, to ensure none gained any moisture before they were weighed again. Since only 4 blocks were weighed at a time, no block would have gained any appreciable moisture in that time.

The basic density was then calculated for each block, i.e.

$$\text{basic density} = \frac{\text{oven dry weight (g)}}{\text{saturated volume (cm}_3\text{)}}$$

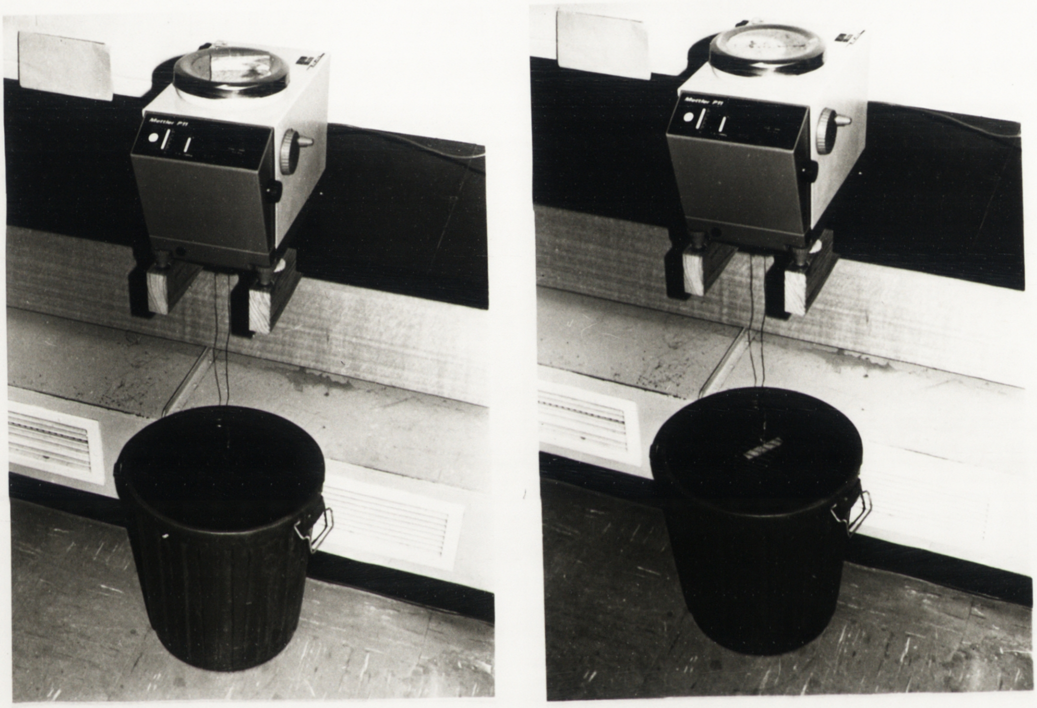

FIGURE 3.2 (a) Weighing Specimen in Air with the Carriage Completely Submerged in Water

(b) Weighing with Specimen and Carriage Completely Submerged in Water

Possible sources of error are:

(a) obtaining the maximum possible saturation with water.

(b) obtaining the saturated weight of the sample in air.

(c) the presence of extractives in the sample and their loss on boiling the block in water (boiling-water extractives).

(d) moisture uptake after oven drying.

Only the (c) source of error had not been taken care of; hence the basic density values had to be expressed on the basis of unextracted wood. Smith (1954) reported the error caused by (b) to be very small and negligible.

3.3 Results

The results of the different groups are shown in Table 3.2 and for the tree selected to show variations with increasing height in Table 3.3.

3.4 Discussion

The results show the basic densities of the unfertilised trees were higher than those of fertilised trees. When the means of the groups were compared using the Student Newman-Keuls (SNK) procedure, they were found not to be significantly different. See Table 3.4

Table 3.2

Basic density of 6-year old *E. globulus* (unextracted wood) taken at breast height.

Treatment	Tree No.	Basic Density (Average)	
A (0.0 kg/ha fertiliser)*	39	0.526	(0.528)
	25	0.530	
	14	0.529	
	2	0.530	
B (188 kg/ha fertiliser	5	0.520	(0.519)
	28	0.526	
	18	0.50	
	41	0.530	
C (565 kg/ha fertiliser)	21	0.52	(0.505)
	33	0.50	
	8	0.50	
	44	0.518	
D (1130 kg/ha fertiliser	12	0.492	(0.490)
	46	0.486	
	34	0.501	
	23	0.480	

* fertiliser Hy-Gold 18 (18% N and 8% P)

Table 3.3

Basic density up the tree selected for this purpose*

Treatment C	Height (m)	Basic Density (g/cm^3)
565 kg/ha	0.25	0.50
(fertiliser)†	1	0.50
	3	0.51
	5	0.56
	7	0.58

* Tree selected was one whose dbh is closest to the mean diameter of treatment C plots, to show the effect of sampling height.

† Fertiliser Hy-Gold 18 (18% N and 8% P)

Table 3.4

Mean basic densities of 6-year old fertilised *Eucalyptus globulus* (unextracted wood) taken at breast height.

Treatment	$\bar{x}$
0.0 kg/ha (fertiliser)	0.528
188 kg/ha (fertiliser)	0.519
565 kg/ha (fertiliser)	0.510
1130 kg/ha (fertiliser)	0.490

Vertical lines indicate that means are not significantly different at P=0.05. The student Newman-Keuls (SNK) procedure was used. This is a stepwise method using the range as the statistic to measure the differences among means.

This agrees with Cromer's (1972) findings with 4-year old *E. globulus* where fertilisation did not decrease the basic density significantly and with Hans *et al* (1972) with *E. grandis*.

Higgs (1969) however found that an application of NPK fertiliser to 28-year old *E. regnans* significantly decreased the basic density of wood produced. The detection of these differences depended on the comparison of basic densities of wood produced before and after fertilisation. The actual densities of the fertilised and unfertilised trees were not significantly different. The detection of such differences as above was not possible in this study since basic density of wood produced before and after fertilisation could not be compared.

Nevertheless a decrease of up to 7.2% was observed between Group A (control) and Group D (1130 kg/ha) and this progressively decreased to 3.4% (Groups A and C) and 1.7% between Groups A and B.

Results from the tree selected to show variations with height reveal that basic density is increasing with increasing height; the heighest basic density being recorded at the 7 metre level. This agrees with the findings of Götze (1964) in *Populus* species and those of Dargavel (1968) in the lower half of the bole of *E. regnans*.

CHAPTER 4

FIBRE LENGTH

4.1 Introduction

Investigations into tracheid dimensions in conifers and those of fibres and vessel segments in hardwoods have been going on for many years mainly because fibre length is closely correlated with the strength properties of paper especially tearing. The longer the fibre length the higher the tear factor.

Hardwoods are now receiving a great deal of attention since they are being accepted as suitable or potentially suitable raw material for pulp and paper. Except in Australia relatively young stands containing a high proportion of immature wood are being harvested and this has been the result of greater use of fertilisers to stimulate growth in an attempt to reduce rotation age.

This section of the study investigates the effect of fertilisers on the fibre length of *E. globulus*, the variations in fibre lengths with height and from pith to bark. Sample trees were selected from plots to which varying amounts of fertiliser had been applied. Again a single tree was also selected and sample discs taken at different heights up the tree.

4.2 Fibre length variations from pith to bark

Dinwoodie (1961) published a detailed review on fibre length. However, much of it dealth with softwoods; very little on hardwoods. However it seems clear that fibre length in the ring nearest the pith is very short (0.1-1.0 mm in hardwoods, then increases in the first few rings after which the rate of increase declines until a maxiumum length is obtained.

Disagreements however exists as to the nature of this increase; whether it is associated with ring number or with linear distance from the pith; and also whether fibre length remains constant or fluctuates after a maximum length has been reached.

Several researchers have recorded that at a given number of rings from the pith, fibre length attains its maximum length, after which it remains constant or very nearly constant in successive rings (Sanio 1872, Bisset and Dadswell 1949). Sanio (1872) who has been cited by practically every subsequent worker, recorded a constant tracheid length in *Pinus sylvestris* after a maximum had been reached between the 25th and 60th rings from the pith. His findings have been supported by Bisset and Dadswell (1949) and Higgs (1969) with *E. regnans*, Dadswell (1958) with *Pinus radiata*, *E. grandis* (Ranatunga, 1964, Bamber and Humphreys, 1963); *E. camaldulensis* (Chudnoff and Tischler, 1963); *E. gomocephala* (Stern-Cohen and Fahn, 1964); *E. deglupta* (Davidson, 1972), *E. virminalis* (Nicholls and Phillips, 1970).

Others disagree with Sanio. Actual decrease in fibre length to the outside of the tree was reported by Hartig (1885) for *Fagus sylvatica*, by Lee and Smith (1916) for *Pseudotsuga* *taxifolia* and by Helander (1933) for *Pinus sylvestris* and *Picea abies*. Dinwoodie (1961) suggests the above observations may be due to the onset of senescence.

Others have reported a slow but steady increase in fibre length after the initial maxiumum has been reached. This was observed by Gerry (1916) for a 255-year old *Pseudotsuga taxifolia*, Kramer (1957) and Jackson and Green (1958b) for 65-year *Pinus taeda* and *P. caribaea* respectively.

Bailey (1914) and Bailey and Shepard (1915) found considerable fluctuation in length after a maximum tracheid length had been reached in *Pinus palustris* and *Abies concolor*. Dinwoodie (1960) also found this fluctuation in *Picea sitchensis* which he tried to relate to ring width.

Elliott (1960) and Dinwoodie (1960) working with Sitka Spruce concluded that the effect of age is only significant in the early years. As a tree matures the effect of age is superseded by that of ring width--which is usually taken as a measure of growth rate. In young plantations an apparent maximum tracheid length occurs between 10-20 years, depending on the species.

4.3 Fibre length variations as related to height up the tree

There is evidence to show that fibre length increases up the tree for a certain distance before decreaseing progressively to the top; the mean at the top usually being less than the mean at ground level. This relationship was first observed by Sanio (1872) for *Pinus sylvestris* and has since been confirmed by others for a wide range of species, e.g. Bisset and Dadswell (1949), Higgs (1969) in *E, regnans*, Desch (1932) in species of *Alnus, Betula* and *Populus*; in *E. grandis* (Ranatunga, 1964); and in *E. deglupta* (Davidson, 1972).

With the exception of the first ring from the pith, tracheid length has been found to increase with increasing height up to some point in the stem, after which the length remained constant. In the ring nearest the pith, tracheid length remained constant or only varied slightly up the stem (Bisset and Dadswell, 1949), Jackson (1959) and Department of Forestry, Queensland (1958). Taylor (1973) reported no significant influence of height on fibre length in *E. grandis* grown in South Africa.

4.4 Relation between cell length and rate of growth

Many attempts have been made to find a possible relationship between cell length and growth rate, using ring width as an index of the latter.

Kuziel (1953) found a negative relationship in loblolly pine (*Pinus taeda* L.). Gerry (1915, 1916) reported no relationship between cell length and ring width in the same species. Vasiljeric (1955) recorded a positive relationship between tracheid length and ring width in European spruce (*Picea abies*, Krast).

Dinwoodie (1960) found a small positive correlation in the youngest 8 rings of Sitka Spruce (*Picea sitchensis*) trees and thereafter a high negative correlation. He related the small positive correlation to the parallel systematic increases outwards of ring width and tracheid length, from the pith. Dinwoodie (1961) in his review believed the balance tended to favour a negative relationship between growth rate and cell length; even though in comparison of trees of different growth rates, considerable differences may be available indicative of a positive relationship between cell length and rate of growth.

Scaramuzzi (1957) compared the fibre lengths in *E. camaldulensis* (syn. *E. rostrata* Schlecht) growing on different sites and concluded they were higher in trees with the slowest growth.

Many studies have shown that increased growth rates have resulted from the application of fertilisers to forest trees. McGregor (1957) found 560 kg/ha of nitrogen alone increased growth rates of slash pine (*Pinus elliottii* Engelm) by 36.6%. Cloud (1950) found an even greater increase in 10-year old slash pine especially for radial growth following the application of 56 kg/ha of ammonium nitrate per hectare. Roth and Evans (1958) concluded that a complete fertiliser augmented by nitrate of soda increased diameter growth more than any of the inorganic nitrogenous fertilisers alone while working with short leaf pine (*Pinus echinata* Mill). However Gilmore and Livingstone (1958) could not find any significant increase in pulpwood volume in slash pine after 19 growing seasons, even though they observed increases in growth rate as a result of NPK application. Klemm (1968) observed a decrease in tracheid length following fertilisation of conifers. Bisset *et al* (1951) observed a decrease in tracheid length of *Pinus pinaster* growing on a site deficient in phosphorus following an application of superphosphate, although it returned to normal after a few years. Gentle *et al* (1968) also observed a slight decrease in the tracheid length radiata pine (*Pinus radiata*, D. Don) growing on a nutrient deficient site following an application of phosphate fertiliser.

Klemm (1968) considered fertilisers had little effect on the tracheid length of European conifers, despite increased cambial activity following their fertilisation.

Possey (1964) observed a decrease of between 11 and 12% in the average tracheid length of 12-16 year old loblolly pine after applying nitrogen, phosphorus and potassium; seven years later the difference was still between 5 and 6%. Zobel *et al* (1961) also observed this decrease in loblolly pine after fertilisation with NPK, though the difference was not significant. Hans *et al* (1972) also found a non-significant decrease in fibre length of *E. grandis* grown in Zambia following fertilisation with NPK.

Higgs (1969) found that NPK fertiliser significantly decreased the fibre length in 28-year old *E. regnans* relative to unfertilised trees.

4.4 Fibre length measurement

Materials

Four trees were selected randomly from each of the four levels of fertiliser applications in the same manner as was done in the density determination. Strips were cut along the radius of each disc taken at breast height from the sample trees. Strips were also taken from the tree selected for height sampling at 0.25, 1, 3, 5 and 7 metres above ground level.

Using the transversing microscope the sample strips were separated into individual growth rings and were marked as to whether they were laid down in the first year and so on. The growth rings were fairly distinct.

4.5 Method

Each piece of wood representing each growth ring laid down each year, was put in a separate test tube. A solution of equal parts by volume of glacial acetic acid and 100 volume hydrogen peroxide was added to each test tube till it was about 3/4 full. The test tubes were then placed in a 500 ml beaker which was then put in a water bath at about 65°C. The test tubes were left over night by which time the wood had bleached white. The macerating solution was decanted and the wood chips were washed with several changes of distilled water. The test tubes were then covered and shaken very gently until most of the fibres had separated into a suspension.

The suspension was stained with 0.5% acid fuchsin and 0.02% safranin left for about 3 hours and then washed again with distilled water. Using a pipette, a drop or two of each suspension was dropped on to clean dry glass slides. A gentle tapping on the underside of the slides ensured a random scattering of the fibres (Hillis - personal communication). The slides were then covered with cover slides and left for a while to ensure that entrapped air bubbles disappeared completely before starting measurements.

Using a projecting microscope the mounted fibres were projected onto a white paper which had been calibrated using a micrometer. A total measurement of 50 whole fibres was made on each slide (one growth ring) giving a total of 300 measurements per tree, using a 15X wide field ocular and a 35X objective giving a 100X projection of the fibres. The microscope used was a Gillet and Sibert conference microscope. (Fig. 4.1).

Results are shown in Table 4.1 (a) and (b) for the group trees and in Table 4.2 for the tree selected for height sampling.

4.6 Discussion

The average fibre lengths of the fertilised trees were less than those of the Control A in all the growth rings examined. This is shown graphically in Figure 4.2 using un-adjusted fibre length data. The difference between Group A (Control) trees and those of Group D (1130 kg/ha fertiliser) is significant at the 95% level using the SNK test in the comparison of means of fibre lengths of the first growth ring. (Table 4.1 (a)). However the differences between Control A, Groups B and C 0.0 kg/ha, 188 kg/ha and 565 kg/ha of fertiliser respectively were not significant in the first growth ring.

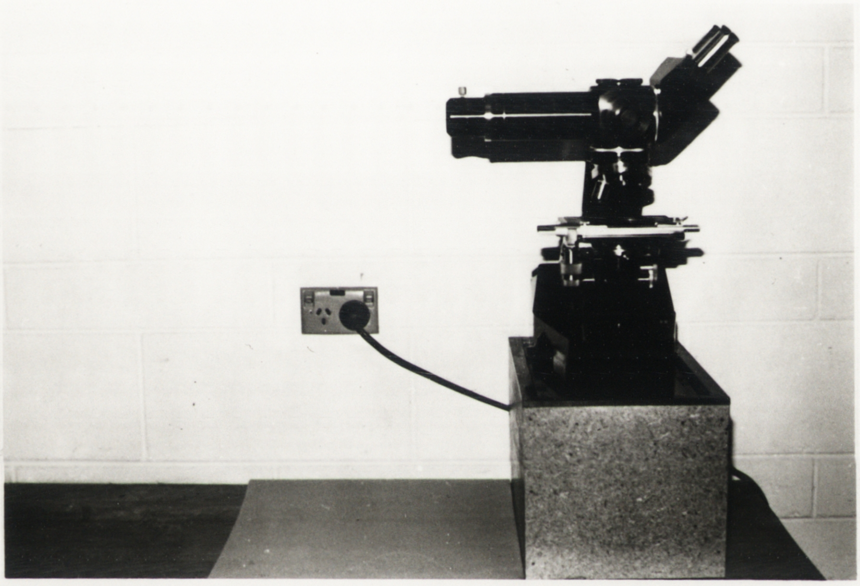

FIGURE 4.1 Gillet and Sibert Conference Microscope

Table 4.1 (a)

Mean fibre lengths in mm of 6-year old fertilised *E. globulus* (four trees per treatment per growth ring)

Treatment	Growth rings fibre length (mm)					
	1	2	3	4	5	6
A (0.0 kg/ha fertiliser)*	.737	.860	.929	1.004	1.06	1.112
B (188 kg/ha fertiliser)	.697	.809	.904	.976	1.02	1.07
C (565 kg/ha fertiliser)	.697	.798	.885	.934	1.00	1.07
D (1130 kg/ha fertiliser)	.625	.749	.846	.932	.991	1.06

Vertical lines indicate non-significant data sets at P=0.05

* Fertiliser HyGold 18 (18%N and 8%P)

Table 4.1 (b)

Mean Fibre Lengths in mm for the Four Different Fertiliser Levels.

Treatment	Fibre lengths
A (0.0 kg/ha of fertiliser)*	0.951
B (188 kg/ha of fertiliser)	0.911
C (565 kg/ha of fertiliser)	0.898
D (1130 kg/ha of fertiliser)	0.868

Vertical line indicates non-significant data sets at P=0.05.

* Fertiliser Hy-Gold 18 (18% N and 8% P)

Table 4.2

Mean fibre lengths of the tree selected for height sampling. The selected tree was the one closest the mean diameter at breast height of Plot 778 of Group C treatment (565 kg/ha fertiliser).

Height (metres)	Growth rings mean fibre Lengths in mm					
	1	2	3	4	5	6
0.25	.630	.783	.847	.905	.986	1.067
1	.640	.767	.779	.903	1.035	-
3	.765	.889	.960	1.03	-	-
5	.781	.885	.988	1.09	-	-
7	.797	1.005	1.19	-	-	-

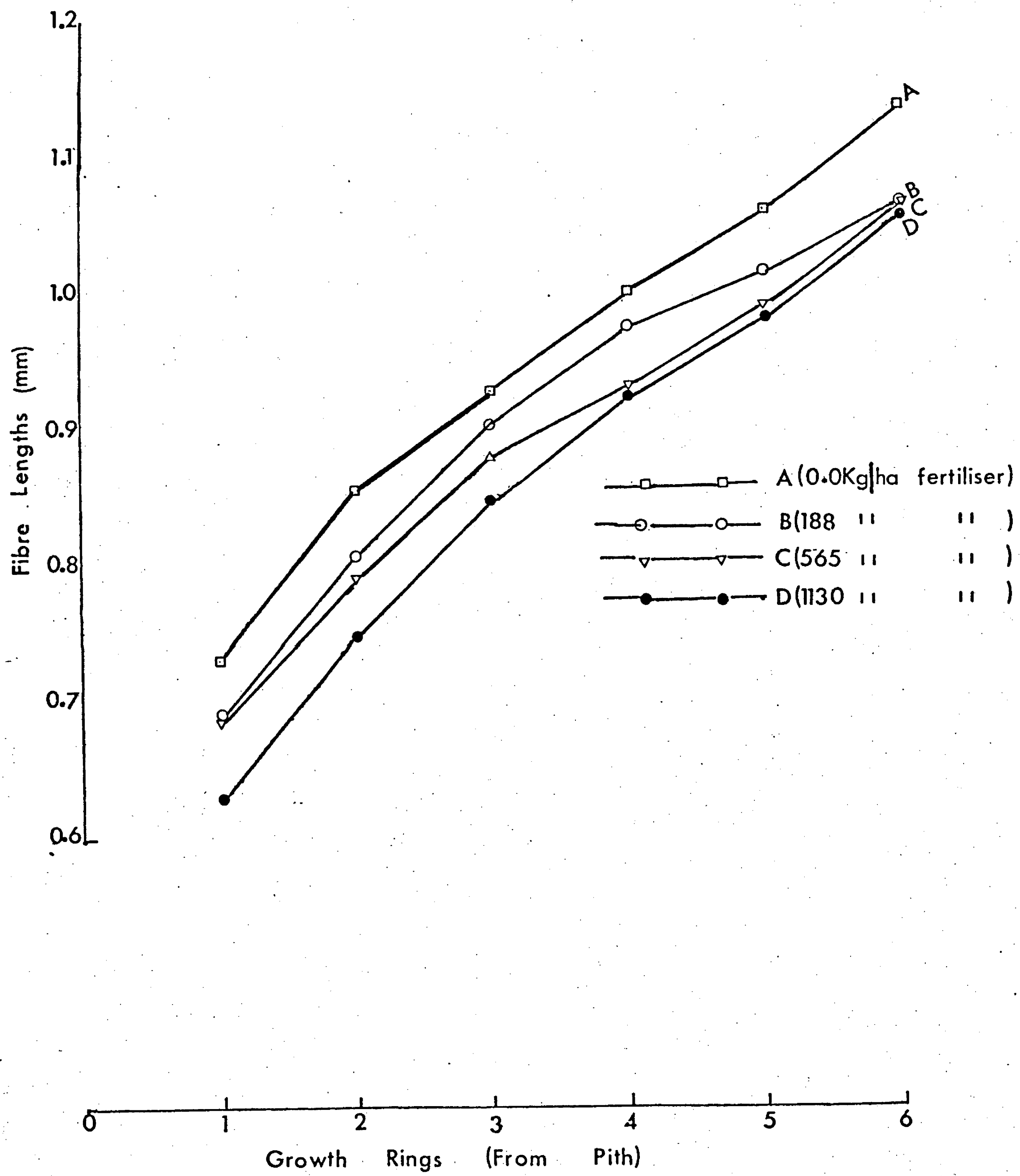

Fig. 4.2. Fibre length variation in different growth rings of fertilised 6-year old *E. globulus*.

However the mean fibre length of the first growth ring of Control A was still 5.5% more than the means of Groups B and C.

In the second growth ring, the mean fibre length of Control A was significantly different at the 95% level from the mean fibre length of the second growth ring of Group D. In this growth ring the mean fibre lengths of Control A, and Groups B and C were not significantly different. Again, however, the mean fibre length of Control A was still 6% longer than that of Group B and 7% more than that of Group C.

From the third growth ring to the sixth growth ring all the mean fibre lengths of Control A and Groups B, C and D are no longer significantly different. But that of Control A is still higher.

The mean fibre lengths for the four levels of fertilisation were not significantly different. (Table 4.1 (b)).

It appears, therefore, that fertilisation only significantly affects the mean fibre lengths of *E. globulus* at the highest fertilisation level D but only in the first two years. As the tree grows older the fertilisation effect decreases and appears to be disappearing in the later years. This agrees with the findings of Elliott (1960) and Dinwoodie (1960) working with Sitka Spruce.

Figure 4.2 suggests that fibre lengths might be the same after a few years but this needs to be tested. Fertilisation does not significantly decrease fibre length of *E. globulus* if a rotation of more than three years is used.

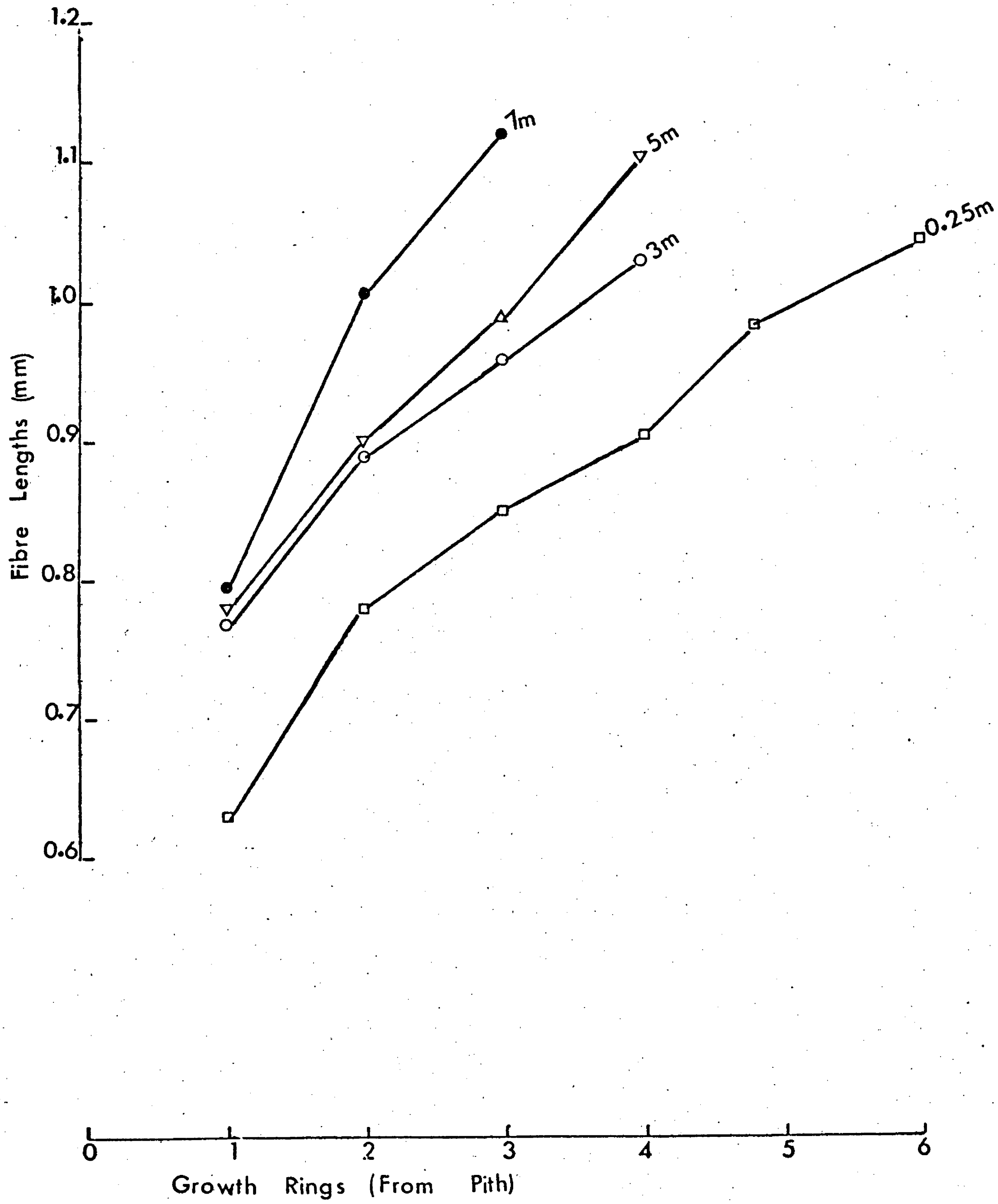

Fig. 4.3. Fibre length variation in different growth rings up the stem of a 6-year old *E. globulus*

This agrees with the findings of Cromer (1972) working with 2-year old fertilised *E. globulus*. He found no significant difference in the mean fibre lengths as a result of fertilisation. Zobel *et al* (1961) showed a considerable but non-significant decrease in tracheid length of loblolly pine (*Pinus taeda* L) after heavy fertilisation.

In the tree selected for height sampling, the increase in fibre length up the stem is noticeable (Table 4.2) and Figure 4.3). The fibre length of the first growth ring nearest the pith varies only slightly but in the other growth rings the fibre length increases sharply. The greatest fibre length is found in the third growth ring at the 7 metre level--in this case the outermost growth ring. In this growth ring a difference of up to 18% is found between it and the same growth ring at the 0.25 metre level. This agrees with the findings of Bisset and Dadswell (1949) working with *E. regnans*.

Even though the increase in fibre lengths of all treatments is gradually slowing down, it is apparent the maximum fibre length is still to be achieved (Fig. 4.2).

Nitrogen fertilisation increases the activity of daughter cells during cambial activity. Since increased rates of multiplicative divisions are usually accompanied by a drop in the mean cell length, a factor such as nitrogen fertilisation which causes an increase in radial growth, should also cause a drop in the mean cell length (Bannan, 1957). From this study, only the higher level of fertilisation gives this effect. However the increased volume of wood produced could more than compensate for this.

Individual tree responses are apparent when individual tree data are scrutinized. Tree 39 from the Control while producing wood of low density had a high initial fibre length of .0787 mm whereas Tree 41 from Group B (188 kg/ha of fertiliser) having a lower initial fibre length of 0.707 mm produced wood of a high density. Tree 21 from Group C produced denser wood and a higher initial fibre length than Tree 18 from Group B (188 kg/ha fertiliser). Such differences in response provide a basis for selecting trees to produce the desired characteristics for the end use contemplated. Zobel *et al* (1961) referred to the extent response to fertilisation can be affected by the individual tree and how this is associated with initial specific gravity but this was not tested in this study.

In conclusion, fertilisation does not have any deleterious effect on fibre length of *E. globulus* if rotation age is 10 years and with lower levels of fertiliser application the early deleterious effect observed in Group D does not show up at all.

CHAPTER 5

FIBRE WALL THICKNESS AND LUMEN DIAMETER

5.1 Introduction

The properties of wood and its derived products often depend on various structural elements of the particular species used. For example, pulp fibres prepared from hardwood species behave differently during paper making from coniferous tracheids and the paper itself will have different properties. This is partly due to differences in cell sizes between the two types of wood. Knowledge of the dimensions of the cells which make up the xylem tissue will give greater understanding of the behaviour of wood either as sawn timber or as raw material for the pulp and paper industry.

Much work has been done to determine fibre lengths (Sanio 1872, Bisset and Dadswell 1949, Hartig 1885, Lee and Smith 1916, Helander 1933 etc) but less has been done on transverse dimensions such as fibre diameter and cell wall thickness (Staffer 1892, Bethel 1943, Lenz 1956, Schulz 1957, Myint Aung 1960, 1962 and Scaramuzzi 1965). That wood structure may vary during the several stages of tree development and that there are definite relationships between anatomical features and wood properties has been reviewed by Dadswell (1958) and Dadswell and Wardrop (1960).

The cell wall thickness of a hardwood contributes to its strength, machinability and pulp quality. The relationships between fibre diameter, lumen diameter and cell wall thickness have an important effect on the flexibility of a fibre during pulping and so affect a number of paper properties (Dinwoodie, 1965).

A number of ratios have also been derived by various investigators in order to correlate the properties of the measured anatomical characteristics with the properties of the wood and reconstituted wood products. The importance of the ratio (usually expressed as percentage) between the diameter of the lumen and the diameter or width of the fibre, called the "coefficient of flexibility" has been fully discussed by Pētēri (1952) in his pulping studies with hardwood species. He found in these studies that provided the average length of fibres was not lower than 7mm, the "coefficient of flexibility" contributed to the tensile strength of hardwood pulps.

Runkel (1952), also working with hardwood species, found that the ratio of double wall-thickness (2W) and lumen diameter (L) of a fibre was important in determining which pulps would have favourable strength properties, and that where the ratio of 2W/L was less than 1, more favourable pulp strengths were usually obtained than when the ratio was more than 1. This ratio has been used as a guide in classifying temperate and semi- and tropical hardwood species especially those of Zaire where growth rate is even throughout the year.

Casey (1960) noted that "fibre coarseness" or the relationship of fibre diameter to length, is at least as important as fibre length in pulp strength developments.

Petroff and Normand (1961) working with tropical woods, established a good relationship between tearing strength and the ratio of fibre length (FL) to fibre diameter (D). This ratio is sometimes referred to as the felting coefficient. Although it has been cited in the technical literature as being associated with paper strength, evidence to support this has been ambiguous in many cases.

Tamolang and Wangaard (1961) found no relationship between fibre length/diam (FL/D) ratio and burst factor, tear factor or breaking length. However, with a modified statistical procedure and the inclusion of more species, Wangaard (1962) found some correlation between this ratio and burst and tear factors.

Watson and Dadswell (1964) concluded that any such correlations between fibre length/diameter ratio and strength properties "are clearly due to a combination of circumstances". In their study of three Eucalypts, they found that *E. regnans* and *E. moluccana* had different paper strengths even though they had similar fibre length/diameter ratios. This will be discussed in Chapter 8 and Table 8.7.

The important arithmetic ratios of the anatomical properties of unfertilised and fertilised *E. globulus* are also presented later in Chapter 8, Table 8.5.

In this section of the study the effect of fertilisation on the anatomical dimensional characteristics of fibres of 6-year old *E. globulus* is investigated.

5.2 Literature review

The literature contains only limited information on the effects of fertilisation on fibre diameter, lumen diameter and double wall thickness. According to Mohr and Roth (1897), Hata (1949), Schultz-Dewitz (1958) and others, the diameter of coniferous tracheids increases with age from pith to bark. However, according to Harlow (1927), the increase with age in the tangential tracheid diamater is less than in other cross-sectional cell dimensions.

Mohr and Roth (1897) in their studies with southern pines, suggested that for most of them, the thickness of a tracheid wall varied inversely with the length of the tracheid. Zobel (1961) working with 16-year old loblolly pine, heavily fertilised at three consecutive annual application rates of 179.4-90.8-90.8 kg/ha of nitrogen (as ammonium nitrate containing 33% of nitrogen), phosphorus (as superphosphate, P_2O_5 basis), and potassium (as potassium chloride K_2O basis) respectively found the reverse, that longer fibres had thicker walls than the shorter. Possey (1964) working with fertilised 16 and 12-year old loblolly pine, fertilised as in Zobel's work just quoted, found that in contrast to all other growth and wood characteristics studied, the tangential diameter of the tracheid failed to show any differences resulting from fertiliser applications. He explained the differences he observed to be due to a normal increase in tangential diameter of the tracheid with age as indicated by Mohr and Roth (1897), Hata (1949) and Schultz-Dewitz (1958).

Possey however found fertilisation caused a significant decrease in radial wall thickness, specific gravity and fibre length as compared with the unfertilised plots in the same study. It is necessary to note here that in the studies of Zobel and Possey, properties of wood formed before and after fertilisation were compared.

Pechman and Wutz (1960) observed a significant decrease in the double wall thickness and specific gravity of fertilised 56-year old spruce. Williams and Hamilton (1961) while reporting a significant decrease in specific gravity, found no significant difference in wall thickness of springwood tracheids of slash pine.

Amos, Bisset and Dadswell (1950) found fibre diameter in *E. delegatensis* was greatest where growth was rapid.

Wooten (1958) working with willow oak (*Quercus phellos* L) found changes in fibre wall thickness and lumen diameter along the stem were exceedingly small and non-significant.

5.3 Materials and method

Four trees were selected randomly from each of the 4 levels of fertiliser applications as in the density determination. They were felled and discs were taken at breast height from each tree. Radial strips were then taken from each disc and from these, small wood blocks roughly 1cm cubes, were cut. A tree was also selected from Group C treatment for height sampling and small blocks of wood taken at the different heights sampled (0.25m, 1m, 3m, 5m and 7m).

Specimens were either used in the green state or saturated with water. The cross-section and tangential surfaces of the blocks were smoothed using either a scalpel or a sliding microtome. The scalpel gave a better surface than the microtome because of its flexibility which allowed a radial "path" to be cut across the annual rings. The slight depression in the surface resulting from this radial "path" could be flooded with water during measurement to ensure that no distortion of images occurs.

Two blocks were taken from each disc, one near the pith, the other near the bark and also at every height sampled in the tree selected for height sampling. The blocks were fully submerged in water and held in position with modelling clay in a small plastic trough. To ensure that air bubbles would not distort the appearance of cell walls, due to the different refractive indices of air and water, the surfaces were frequently washed with an aspirator.

The optical contrast between cell walls and lumina was increased by staining with a 1% aqueous mixture of equal parts by weight of malachite green and methylene blue. Since a transverse cut on a sample intersected the fibres randomly along their lengths, measurements of contiguous fibres in the radial direction gave reliable means provided the tips of fibres were excluded in the measurement. Such contiguous rows of fibres were randomly selected for measurement.

The instrument used was a Leitz dual linear traversing microscope (Fig. 5.1). A 55X water immersion objective, a 1.25X intermediate stage and 10X stereoscopic ocular with cross hairs were used giving an overall magnification of about 687X. Illumination was provided by a high intensity Xenon arc lamp. The advantages of this instrument and method over other known methods have been summarised by Smith (1965).

Because of the poor alignment of the fibres tangentially as compared with radially, the samples had to be reorientated frequently to measure fibre wall thickness. It was however easier to measure wall thickness in the radial direction because the line of traverse could be reorientated though the mid-point of successive tangential walls at right angles in any selected radial file. To change the orientation, a mechanical stage with adjustment along two axes at right angles was superimposed on the micrometer traversing table. Adjusting this stage did not alter the micrometer scale drums. The microscope unit travelled to the right, the stage to the left, along the micrometer screws, with the image travelling from left to right in the viewing field. The specimen could then be reorientated in any direction and measurements made with the cross-hairs of the ocular oriented horizontally. If by-passing or offsetting had to be done to find a new radial file of fibres without any obstruction, the mechanical stage was moved forwards or backwards to bring a new radial file into view. Anatomical elements by-passed during measurement included vessels, diffuse and vasicentric Parenchyma and the obvious tips of fibres. The narrow rays did not appear to influence the

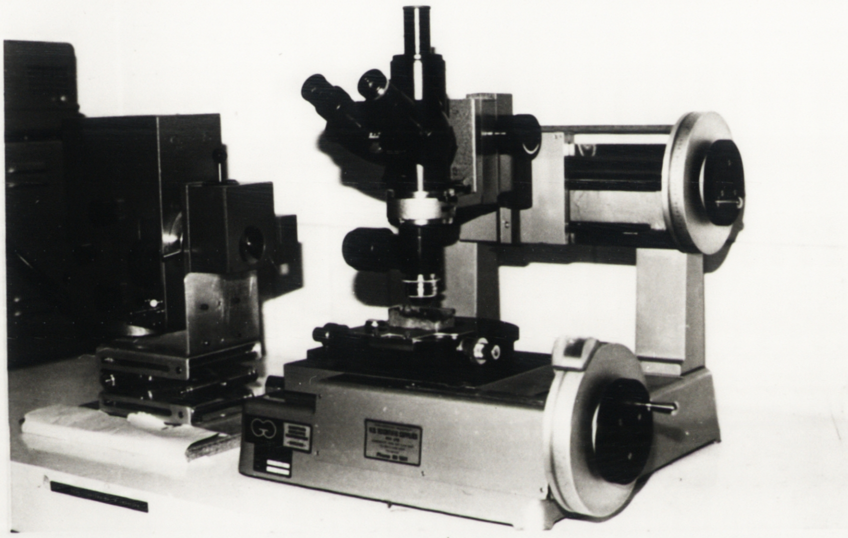

FIGURE 5.1 Leitz Dual Linear Traversing Microscope

dimensions of adjacent fibres.

The advantage of the instrument is derived from its dual linear scales enabling the combined radial width of the double cell walls to be accumulated on one scale and the combined radial widths of lumina on the other.

A total of 100 measurements were made on every sample, 50 of double wall thickness and 50 of lumen diameter, and average fibre dimensions were determined. Results are shown in Tables 5.1 (a) for the group trees and 5.2 for the tree selected to show the effect of sampling height.

5.4 Analysis of result

As growth rate increased the mean double wall thickness decreased and the mean lumen diameter increased (Table 5.1 (a)). The mean double wall thickness of the Control A was not significantly different from that of Group B (188 kg/ha fertiliser). However that of the control was significantly different at the 95% level from those of C (565 kg/ha fertiliser) and D (1130 kg/ha fertiliser). Group B was however not significantly different from Groups C and D (Table 5.1 (b) SNK analysis).

Fertilisation therefore decreases the mean double wall thickness even though at the lower level of fertilisation (188 kg/ha) the decrease is not significant.

The mean lumen diameter of the control and Group B was not significantly different while at the higher levels of fertilisation of C and D, the mean lumen diameter was

TABLE 5.1 (a)

Mean double wall thickness (2W) and mean lumen diameter (L) of 6 year old fertilised *E. globulus* in micrometers.

Treatment and Tree Number	2W	Treatment Mean	L	Treatment Mean
A(0.0 kg/ha fertiliser)				
25	5.7		10.2	
39	5.6	5.53	10.1	10.1
14	5.4		10.2	
2	5.4		9.9	
B(188 kg/ha fertiliser)				
18	5.2		10.5	
28	5.3	5.23	10.3	10.45
5	5.2		10.5	
41	5.2		10.5	
C(565 kg/ha fertiliser)				
21	5.1		10.7	
44	5.1	5.00	10.5	10.78
8	5.1		11.1	
33	4.7		10.8	
D(1130kg/ha fertiliser)				
34	4.5		12.1	
46	4.8	4.93	11.9	12.03
12	5.2		12.5	
23	5.2		11.6	

TABLE 5.1 (b)

Mean double wall thickness (2W) and mean lumen diameter (L) of 6-year old fertilised *E. globulus* trees. Sample blocks were taken at breast height.

Treatment	Double wall thickness (2W) in micrometers	Lumen diameter (L) in micrometers
0.0 kg/ha (fertiliser)	5.53	10.1
188 kg/ha (fertiliser)	5.23	10.45
565 kg/ha (fertiliser)	5.00	10.78
1130 kg/ha (fertiliser)	4.93	12.03

Vertical lines indicate non-significant data sets at P=0.05.

TABLE 5.2

Mean double wall thickness (2W) and lumen diameter (L) for the tree selected to show effect of sampling height.*

Height Metres	Double wall thickness 2W (micrometers)	Lumen diameter L (micrometers)
0.25	4.92	10.71
1	4.86	10.6
3	4.80	10.54
5	4.78	10.48
7	4.64	10.36

Vertical lines enclose non-significant subsets at P=0.05.

* The tree chosen was the one closest to the mean diameter at breast height of the Group C treatment plots. To enable corresponding radial positions to be compared, only the three growth rings nearest the pith were sampled.

significantly higher than that of Groups A and B. It would appear that the lumen diameter is more sensitive to higher levels of fertilisation than the mean double wall thickness (Table 5.1 (b)).

In the tree selected for height sampling, the mean lumen diameter and the mean double wall thickness were found to follow the same trend in the corresponding positions up the stem. The double wall thickness and lumen diameter were greatest, but not significantly so at the 0.25 metre level. Even though there was a slight decrease in both parameters up the stem, there was no significant difference between any of the levels from the 0.25 metre level to the 7 metre level, the actual difference being only a few micrometres.

5.5 Conclusion

Fertilisation significantly decreased double wall thickness and increased lumen diameter of the higher levels of fertilisation of 565 kg/ha and 1130 kg/ha of fertiliser. However at the lower level of fertilisation (188 kg/ha) the lumen diameter was not significantly different from the Control Group A whereas the double wall thickness while not significantly different from Control A was also not significantly different from the two higher levels of fertilisation. Still the double wall thickness of Group A was significantly different from that of Group D (1130 kg/ha). It would appear that at the lower level of fertilisation lumen diameter is less sensitive to fertilisation response than double wall thickness. This agrees with Possey (1964) and the difference in response,explainable in terms of age (Possey, 1964); the lumen diameter increase is partially due to age. It also agrees with Amos, Bisset and Dadswell (1950) working with *E. delegatensis* where they found an increase in fibre diameter where growth is rapid. Fibre diameter is a combination of the double wall thickness and lumen diameter, the net effect of fertilisation as can be seen in Table 5.1 (b) is greater fibre diameter.

Double wall thickness was found to be greater in long fibres than in short. Mohr and Roth's suggestion that the thickness of tracheid wall varied inversely with length of the cell could not be substantiated in this study.

The results also agree with those of Zobel *et al.* (1961), working on fertilised loblolly pine.

In the tree selected to show the effect of sampling height, fertilisation did not significantly affect the double wall thickness and lumen diameter up the stem. This agrees with Wooten (1958) working with oak, who found the changes in fibre wall thickness and lumen diameter were exceedingly small and non-significant.

CHAPTER 6

VOLUMETRIC PERCENTAGES OF TISSUES IN 6-YEAR OLD *E. GLOBULUS*.

6.1 Introduction

The types, frequency and distribution of anatomical elements in wood influence its physical properties. The proportion of ray tissue for example could possibly affect the extent of transverse shrinkage and swelling (Kelsey, 1963). Ray tissue also influences the radial permeability of wood, since it, together with vessels, is the most effective pathway for radial movement of liquids (Côté, 1963; Wardrop and Davies, 1961).

Vessels also have an important effect on the surface properties of paper. Because of their shape, they do not bond well with fibres and when present on the surface tend to cause picking during printing (Colley, 1975). The size, area and the ratio of vessel volume to fibre volume are important factors in this problem. However the recent work of Colley and Ward (1976) shows that some eucalypt vessels had a greater inherent tendency to cause picking problems, *E. deglupta* being an example.

Parenchyma cells while not contributing to paper strength, allow more compact sheets to be formed and might thus contribute to interfibre bonding (Hillis, 1975).

The distribution of tissues has an important effect on basic density; a most useful indicator of the value of a pulpwood for a particular use (Wangaard, 1958). However the relationship between basic density and anatomical characteristics has been examined to a limited extent only in angiosperms.

In this section of the study, the volume of tissue types is investigated, and how this is affected by the application of fertilisers.

6.2 Literature Review

The information on the relationship between tissue distribution and basic density is only scanty but also somewhat contradictory and more quantitative data are required. The only known attempt is that of Bethel (1943) when he carried a quantitative investigation on relation of wood density to percentage summerwood in young chestnut oak (*Castanea dentata* Borkh). He found a closely related linear relationship between the two.

Stauffer (1892) found density variation in the vertical direction was influenced by the proportion of vessels in the stem of beech (*Fagus sylvatica L*). However Schulz (1957) found vertical density variations in beech to be associated with variation in fibre volume and the same was found to be true in oak (*Quercus spp*) by Pechman (1958), whilst in ash (*Fraxinus excelsior L*) Pechman (1958) found fibre wall thickness to be the principal factor influencing wood density variation. Lenz (1956) working with white willow (*Salix viridis* Fr) found variations in density due to the variation in the frequency and distribution of various elements. Scaramuzzi *et al* (1963, 1964) found a close linear relationship between basic density and transverse fibre dimensions. Fibre wall thickness had the strongest influence, followed by fibre diameter, the two factors accounting for 90% of the variation. The proportion of wood volume occupied by fibres had no significant effect on the density variation of many eucalypts he studied.

In the studies of the differences in the basic densities of *E. albens* and *E. camaldulensis* each with percentages by volume of fibres, vessels and parenchyma of 68, 18, 14 and 58, 17 and 25 respectively, Hillis (1975) assigned the difference mainly to the different proportion of fibres since both species had a Runkel ratio (2 W/L) of less than 1. Chudnoff *et al* (1963) found the combined effect of the proportion of wood volume occupied by fibres and their cell wall proportions accounted for almost the total variation in the basic density along the stem radius of *E. camaldulnesis* from which extractives had been removed.

Myer (1922) also reported that wood with high specific gravity had more ray volume than that of lower specific gravity. Harlow (1927) reported pronounced variations in ray volume between sites. Goggan (1961) believed that heritability had a prominent influence in determining ray volume. Taylor (1966) observed differences in ray volume from plot to plot in yellow poplar. Also in his recent work Taylor (1973) reported that specific gravity was not influenced greatly by ray volume in *E. grandis*.

grown in South Africa. He observed that specific gravity increased as the wall thickness and the fractional wall volume increased. Specific gravity was also found to increase when there was an increase in fibre volume and decreased when parenchyma volume increased.

Very little has been reported on the variation in the relative proportion of tissue types although in many instances mature trees up to 300 years old have been considered. The results with respect to relationships between age and distance from the pith and the relative proportions of tissue types have also been conflicting.

Jayme *et al* (1943), Linnemann (1953) and Ishida et al (1963) found no significant relationship between the relative proportions of tissues by volume and age or distance from the pith. Schulz (1959) and Courtois *et al* (1964) found that fibre volume proportion decreased and vessel volume proportion increased with increasing distance from the pith.

Taylor (1968) found vessel volume in yellow poplar increased at a more or less uniform rate from pith to bark; increased significantly up the stem; and was strongly correlated with ring width. He also found the trees with the highest vessel volume had the lowest specific gravities and that ray volume differences were quite small between trees but sharply decreased with height from breast height to about 7 metre followed by a slight decrease with successive heights in individual trees. In his recent work on *E. grandis* grown in South Africa Taylor (1973) reported large variations in proportion of tissue types.

He could not observe any obvious pattern of tissue type variation with height. The variation of volume of fibre from pith to bark at every sampling point was small. There was a slight decrease in the volume of longitudinal parenchyma and vasiocentric tracheids as distance from the pith increased. However he observed the volume of vessels and rays appeared to be almost constant throughout the radial growth zones.

Large diameter fibres were found to occur in xylem tissue with a large proportion of fibre volume and a reduced amount of vasiocentric tracheids and parenchyma (both axial and ray parenchyma).

He found general variations within geographical areas were greater in most cases compared with differences between geographic areas.

Scaramuzzi (1955) found little variation in the volume proportions of wood elements in a single *Populus euramericana* Dode tree from a ten year old plantation.

Wooten (1968) working with willow oak found the greatest volume percentage of ray and vessel tissues generally occured at or beyond the sixth growth ring from the pith; minimum values for these types consistently at the second growth ring from the pith whilst the greatest proportion of fibre volume was adjacent to it.

Nicholls and Phillips (1970) working with *Eucalyptus viminalis* could not find any systematic variation for the percentage by volume of vessels and fibres either from pith to bark or with age or age within a class locality. However they observed that in the same age class and nominally equivalent growth rings, the coppice material tended to produce a greater proportion of fibres than the seed propagated material. This they thought needed some further investigation, so as to know the relative incidence of tension wood in both material types. Onaka (1949) reported that the presence of tension wood markedly reduces the number and size of vessels and hence a general reduction in the percentage volume of vessels.

Davidson (1972) working on *E. deglupta* found no clear systematic variation of percentage volume of fibres and rays because the variation patterns were very complex. He however reported a greater proportion of vessel volume in the outer layers of the upper part of the trunk compared with the lower near the pith.

6.3 Materials and Method

The sampling procedure for wood blocks was as given in Chapter 3 in wood density determination. Radial strips were taken from each block and from these roughly 1 cm cubes were cut half way between the pith and the bark. The cubes were either in the green state or had been saturated with water. The cross-section and tangential surfaces of the cubes were smoothed off using either a scalpel or a sliding microtome.

6.3.1 Method

Ray volumes were measured on the tangential surface of the cubes while the proportions by volume of other tissue types were determined on the cross-section surface. The volumes of the other tissue types were adjusted (see later) to remove the discrepency in ray volume between the two surfaces on which measurements were carried. This is because rays, once formed run continuously in the direction from pith to bark; therefore the percentage by area measured on the tangential surface is equivalent to the percentage by volume (Smith, 1965).

The contrast between cell wall and lumen was increased by staining in a 1% aqueous solution of equal parts by weight of malachite green and methylene blue. The instrument used was a Leitz dual linear traversing microscope. See Figure 5.1. A 55x or 22x water immersion objective, a 1.25x intermediate stage and a 10x stereoscopic ocular with cross hairs were used. Illumination was provided by a high intesnity Xenon arc lamp. The advantages of this instrument compared with others have been summarised by Smith (1965).

In determining the percentage of rays on the tangential surface of each cube, one traverse at right (Figure 5.2) angles tothe rays was considered enough. The ray readings were accumulated on one scale and the non-ray readings on the second scale. The surface was also stained with the 1% staining solution referred to above. At the end of the traverse, the accumulated readings were summed up.
Care was taken to zero the two scales at the start of each

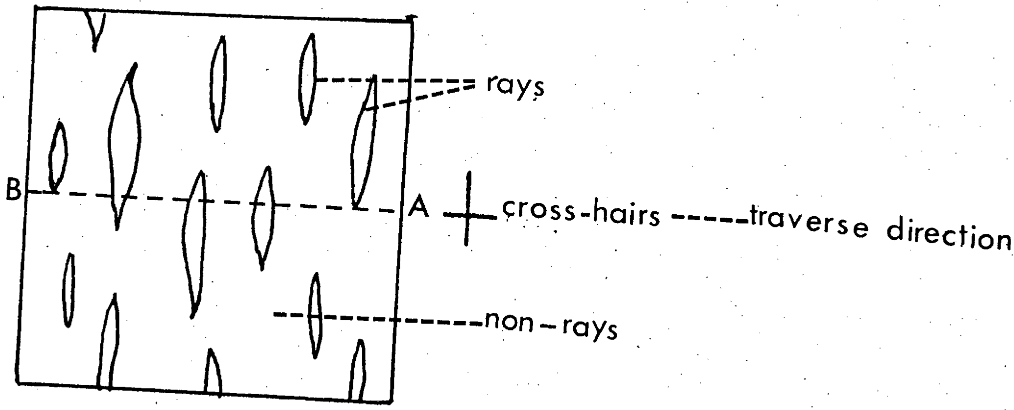

FIG. 5.2

Diagramatic representation of the tangential surface of wood sample of *E. globulus*.

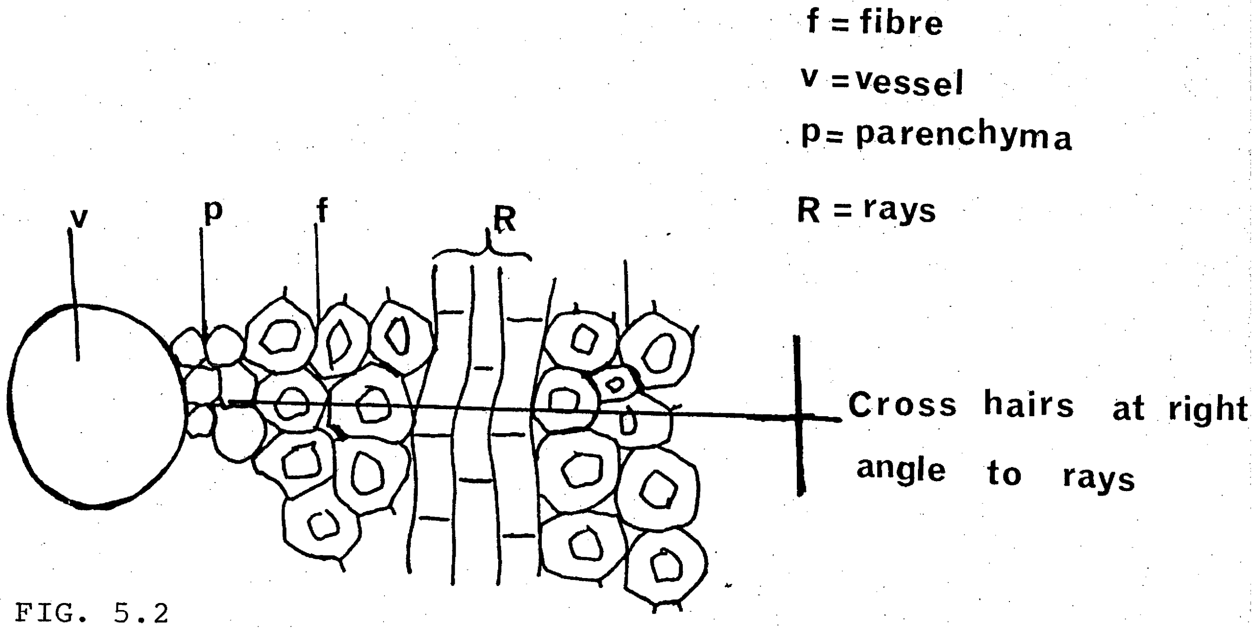

FIG. 5.2

Diagramatic cross-section of *E. globulus*.

traverse measurement.

Rays x from Scale 1

Non rays y from Scale 2

Thus ray % = $\frac{x}{x+y} \times \frac{100}{1}$ (Z%)

On the cross-section surface a 22x objective was used and the traverse was still placed at right angle to the different tissues. See Fig. 5.3. One scale was used to accumulate fibre tissues: on the second scale different readings were recorded for the different tissues as they were traversed. Ten traverses were taken on each surface. At the end of each traverse the accumulated reading on Scale 1 for fibres was read.

Movement of Scale 2	Element Type	Element Width (u)
0		
40	Ray	40
60	Parendyma	20
180	Vessel	120
210	Ray	30

Total Measurements

Element	Length (u)	% of cross-section
from Scale 2		
Ray	1500	15
(∴ Non-ray		85)
Parendyma	500	5
Vessel	2000	20
From Scale 1		
Fibre	6000	60

Since rays had already been measured on the tangential surface, the above figures were then adjusted for percentage of non-rays as follows:

From tangential surface

% Rays = Z%

% Non-rays = (100-Z)%

Adjusted volume percentage of non-rays =

$$P = \frac{5}{85} \times \frac{100-Z}{100}$$

$$V = \frac{20}{85} \times \frac{100-Z}{100}$$

$$F = \frac{60}{85} \times \frac{100-Z}{100}$$

TOTAL R + P + V + F = 100%.

Specimens were held in a plastic trough with modelling clay and flooded with water. The only precaution necessary was to ensure that all surface air bubbles were removed by flushing specimens constantly with an aspirator.

6.4 Results

The results are set out in Table 6.1 for the group trees and 6.2 for the tree selected for height sampling. Table 3.8 summarises the results from Table 6.1.

6.5 Analysis of Results

The data for the group trees and those for the tree selected for height sampling were subjected to SNK analysis for the differences in the means (see Tables 6.2

and 6.3).

The percentages of fibre, ray parenchyma and vessel volume were not significantly different between the groups although fibre volume showed an increase with increasing levels of fertilisation. The large diameter fibres were associated with larger volumes of percentage fibres and reduced amount of volume of parenchyma especially in the highest level of fertilisation.

The percentage volume of fibres and vessels increased with decreasing basic density while decreasing cell wall thickness was associated with higher volume of fibres and decreasing basic density.

According to the work of Kellogg and Ifju (1962), the proportion of fibre wall to total wall for hardwoods within specific gravity range of 0.222 to 1.180 g/cm^3 fallswithin 67 to 89 percent with the exception of very low density wood such as *ceiba* species. It thus appears that low density woods, having very thin-walled fibres would require a high fibre volume in order to reach this value, whereas high density woods, having thick walled fibres, could have a much lower value. It is not surprising therefore that the Control A with a higher basic density had a lower volume of fibre compared with Group D (1130 kg/ha) with its lower basic density and thin-walled fibres. Lumen diameter was negatively correlated significantly with basic density at the 5% level of probability (Table 6.4). Double wall thickness was also correlated significantly though negatively with the percent fibre volume at the 5% level.

For the tree selected for height sampling, the variation of fibre volume was along the pattern of the density variation, higher fibre volumes occured where basic density was high. However there was no significant difference between the percentage fibre volume for the heights sampled.

The percentages of parenchyma volume for the first three sampling points (0.25, 1 and 3 metre) were not significantly different but these were significantly different from those at heights 5m and 7 metre. These last two sampling heights were however not significantly different from one another.

6.6 Conclusions

Fertilisation did not have any strong effects on the volume percentages of cell types. The fibre and vessel volumes increased with fertilisation level though the increases were not significantly so.

Double wall thickness was significantly correlated with fibre volume, negatively at the 5% level of probability.

The variations of fibre volume with height were not significant but the vessel and parenchyma volume percentages were significantly affected by height.

It appears more trees need to be selected for height sampling to get a clear trend of the variations of percentage volume of cell types with height.

TABLE 6.1

Volumetric percentages of cell types in the wood of 6-year old fertilised *E. globulus*. (Breast height samples)

tment and number	Cell type and volumetric tissue percentage			
	Fibre	Ray	Parenchyma	Vessel
.0 kg/ha fertiliser)				
2	69.63	11.88	7.57	10.92
39	70.15	12.40	7.36	10.09
14	69.56	11.78	8.49	9.03
25	69.70	11.90	8.40	10.14
age ($\bar{x}$)	69.76	11.99	7.96	10.01
88 kg/ha fertiliser)				
5	70.07	11.70	6.40	11.83
28	70.66	11.14	7.28	10.92
18	69.84	12.02	6.94	11.20
41	70.68	11.64	7.48	10.20
age ($\bar{x}$)	70.31	11.63	7.03	11.04
65 kg/ha fertiliser)				
21	70.20	11.48	7.29	11.03
33	72.19	12.15	6.56	9.10
8	70.12	11.08	7.30	11.50
44	71.03	10.31	7.42	11.24
age ($\bar{x}$)	70.88	11.26	7.14	10.72
130 kg/ha fertiliser)				
46	70.70	10.61	6.03	10.66
23	71.13	11.78	6.44	10.65
12	71.02	10.12	7.61	11.25
23	72.89	11.55	7.00	8.56
age ($\bar{x}$)	71.44	11.01	6.77	10.28

TABLE 6.2

Percentage of cell types in a 6-year old *E. globulus* tree from Treatment C (565 kg/ha) selected for height sampling.

mple height (n metrics)	Cell type volumetric percentages			
	Fibre	Ray	Parenchyma	Vessel
0.25	68.02	13.75	9.45	8.78
1	68.24	13.06	9.32	9.38
3	69.62	12.14	8.49	9.75
5	70.83	12.12	6.78	10.27
7	71.90	12.08	5.86	11.16

The results within the brackets are not significantly different at P = 0.05 (SNK test).

TABLE 6.3

Volumetric means of cell types in the wood of a 6-year old fertilised *E. globulus* expressed as percentage.

Treatment	Cell types (volume in %)			
	Fibre	Ray	Parenchyma	Vessel
A (0.0 kg/ha fertiliser)	69.76	11.99	7.96	10.01
B (188 kg/ha fertiliser)	70.31	11.63	7.03	11.04
C (565 kg/ha fertiliser)	70.88	11.26	7.14	10.72
D (1130 kg/ha fertiliser)	71.44	11.01	6.77	10.28

The results within the brackets are not significantly different at P = 0.05 (SNK test).

TABLE 6.4

Summary of statistics of linear regression Y = a-bX of some anatomical wood characteristics and basic density and fibre volume of *E. globulus* (Y = basic density and percentage fibre volume and X = anatomical characteristics as shown in the Table).

Property	Number of observations	a	b	Correlation r	Coefficients r^2
Basic density X Double wall thickness	4	0.216	0.057	0.907	0.823
B.d X lumen diameter	4	0.730	-0.020	-0.987*	0.974
B.d. X fibre diameter	4	0.909	-0.025	-0.920	0.847
Fibre volume (F.V.) X Double wall thickness	4	22.765	-0.250	-0.981*	0.962
F.V. X Lumen diameter	4	-68.609	1.125	0.934	0.872
F.V. X Fibre diameter	4	-36.889	0.749	0.824	0.678

P = 0.05
r.0.05 = 0.950

CHAPTER 7

EXTRACTIVES IN FERTILISED 6-YEAR OLD *E. GLOBULUS* AND THEIR INFLUENCE ON PULP AND PAPER PROPERTIES.

7.1 Introduction

The shifting of emphasis from extensive to intensive management of forests in many parts of the world; the use of fast-growing species; the use of fertilisation of unfertile soil and fertile ones, to reduce the rotation age, inevitably means that a large proportion of world's future industrial wood supply, will come from young plantations. This is all in an attempt to meet increasing world wood demand which is already exceeding supply in some parts. Also a proportion of wood supply will have to come from previously unexploited sources such as tropical and sub-tropical forests.

In the past wood anatomists have concentrated on the structure of wood, while paying less attention to the non-structural properties especially in the pulp and paper industry. This is probably understandably due to the fact that the traditional favoured species did not contain so high amounts of non-structural materials as to impose limitation on their uses. The changing types of raw materials now creates the need to investigate their chemical properties, since a lot has already and still being done to control the quality of plantation grown material through breeding.

Uniformity is usually desirable in relation to wood quality where aesthetics are not involved. One non-structural aspect of wood tending towards non-uniformity is the variation in the amount and type of extractives present. Their presence, distribution and physical and chemical nature may have an important influence on uses to which wood may be put. Extractives are of many kinds and include calcium salts, waxes, oils, kinos, silica, gums, resins, tannins, starches, alkaloids, various volatile substances and colouring matter.

Extractives are non-structural or secondary constituents of plants. Usually they can be removed with neutral solvents, although dilute alkali may sometimes be needed. A large number of the components of extractives from different trees that have been isolated and identified, have been found to represent many kinds of organic compounds. The polyphenolic compounds which do not include lignin, are the most common components. There are also tropolones, fats, resins, carbohydrates such as arabinogalactan, terpenes etc. The amount of extractives in wood is dependent on several factors especially the species, the age and the amount of heartwood. Extractives are found mainly in parenchymatous tissues, but can be found also in vessels and fibres and in a few cases, in specialised cells.

Extractives are formed normally in heartwood or, after injury, insect or fungal attack, in sapwood. The latter differ from normally formed extractives in some species. In addition there could also be differences in composition between extractives in heartwood and sapwood of the same species, although according to Hillis (1971) the differences are not very great in some eucalypt species.

In eucalypts, extractives are mainly polyphenolic, large amounts of which can be present in the heartwood of some species. More is found in the outer than the inner heartwood. They are also present in small but significant amounts in the sapwood. Hillis (1969) found five or more percent of the oven-dry weight of wood may consist of extractives in some eucalypt heartwoods.

The use of eucalypt woods with a high polyphenol content for pulping has introduced a number of new problems to the pulping industry. However the quality of such eucalypt wood varies, as it may come from over mature trees with high polyphenol content, from fire and insect damaged trees containing kino pockets or veins or recently from fast grown regrowth trees with a low polyphenol content. Polyphenols of different kinds lower yield of pulp, increase chemical consumption during pulping, reduce permeability, increase bleaching requirements and adversely affect chemical recovery.

In this section the amount of extractives in 6 year old fertilised *E. globulus* wood and bark will be determined and the effects these have on pulp and paper made from wood only and wood with bark added. The pulping was done by the staff of A.P.M. Limited, Research Laboratories, Victoria.

7.2 Literature review

Forests are fertilised to improve growth on poorer sites and to increase that on the more fertile sites, thereby reducing rotation age. Such treatments must include a knowledge of whether or not they cause important changes in the chemical composition and the physical properties of wood. A comprehensive bibliography with abstracts has been compiled by White and Leaf (1957) and more recently by Mustanoja and Leaf (1965) covering 1957-1964.

The response to fertilisation of Douglas fir 10-15 years old was studied by Gessel et al (1951) and Gessel and Walker (1956); and of 30-year old trees of the same species by Shareeff (1955) and Gessel and Shareeff (1957). The rate of growth and composition of quaking Aspen wood (*Populus tremuloides* Michx.) and large tooth Aspen (*Populus grandidentata* Michx.) in relation to natural soil fertility have been studied by Wilde and Paul (1948, 1951). These studies concentrated on the physical properties changes of wood and paid less attention to chemical changes, arising from fertilisation.

Erickson and Lambert (1958) found the amount of extractives in 30-year old Douglas fir following fertilisation was significantly different from control plots at the 95% level. The fertiliser composition and rates of application were: 336 kg/ha of nitrogen; 224 kg/ha in ammonium phosphate and 112 kg/ha in urea; 168 kg/ha of phosphorus (P_2O_5 basis); 34 kg/ha of Potassium (k_2O basis); and 56 kg/ha of lime. P, K and lime were applied in the first year only; the nitrogen during a four year period.

They concluded the increase in extractives was due to the increased amount of those soluble in alcohol-benzene occuring in the sapwood (the last four growth rings) as a result of fertilisation. They also found the amounts of ether and hot water soluble extractives were not significantly affected by fertilisation.

Kurt (1948) using the same species, fertiliser and rate of application as Erickson and Lambert (1958), found greater amounts of both alcohol and ether soluble extractives. However, his samples came from a different site to those of Erickson and Lambert and from older trees with a lot of heartwood.

Cromer and Hansen (1972) working with wood from 2-year old fertilised *E. globulus* from the same experiment as trees that are used in this study, found the amount of both boiling water and alcohol soluble extractives was not significantly different from that in control trees following fertilisation with HyGold 18 (the composition and rate of application are shown in Table 3.1).

7.3 Materials and method

The original materials were as for the density determination in Chapter 3. Wood blocks were cut from each disc taken at breast height from the sample trees grown at 4 different levels of fertilisation and from the tree selected for height sampling, wood blocks were taken at the different heights sampled. The wood samples were then split into match stick sizes with a pocket knife and a chisel. They were air-dried for about 3 days and then ground in a small Wiley mill fitted with 40mm mesh screen. The ground samples were then dried again, this time in a vacuum oven at a temperature of 50°C for about 3 days; 50°C being used to avoid loss of volatile extractives. Sub-samples weighing between 4.9-5.0 gm were taken from each sample. The same method was used in preparing the bark for extraction.

7.4 Extraction

Whatman extraction thimbles were first weighed empty and then with wood samples in them. The samples were extracted in a Soxhlet apparatus first with diethyl-ether for 16 hours, then with methanol for another 16 hours. The extracts were then poured into dried and weighed 50ml beakers and put in a fume cupboard to allow the solvent chemicals to evaporate. The extracts were then dried for 3 hours at 50°C and finally weighed. The yields were expressed as percentage of the oven-dry weight of the wood. The same method was applied to the bark samples. A few beakers at a time were transferred to a desiccator and later wieghed. This procedure was necessary to avoid moisture absorption.

Results are shown in Tables 7.1 (a) for sample trees and 7.1 (b) for the tree selected for height sampling.

TABLE 7.1 (a)

Extractives in 6 year old fertilised *E. globulus* wood and bark in percentage of the ovendry weight of samples, extracted in the order listed.

Treatment and Tree Number	Wood		Bark		Total	
	Ether	Methanol	Ether	Methanol	Wood	Bark
A (0.0 kg/ha fertiliser)						
39	1.21	2.95	2.65	6.84	4.16	9.49
25	1.40	1.89	1.89	6.51	3.29	8.40
14	1.21	1.77	2.75	7.45	2.98	10.17
2	1.45	1.49	1.51	6.50	2.94	8.01
Average ($\bar{x}$)	1.32	2.03	2.20	6.82	3.35	9.02
B (188 kg/ha fertiliser)						
5	1.14	2.04	2.32	6.79	3.18	9.11
28	1.89	2.19	2.39	7.23	4.08	9.62
18	1.02	2.26	2.75	7.16	3.28	8.91
41	1.37	2.21	3.60	7.28	3.58	10.88
Average ($\bar{x}$)	1.36	2.18	2.77	6.86	3.53	9.63
C (565 kg/ha fertiliser)						
8	1.09	2.33	2.12	7.24	3.42	9.36
21	1.25	2.08	2.01	7.82	3.33	9.83
33	1.85	2.66	3.29	7.09	4.51	10.38
44	1.44	1.76	2.84	6.17	3.20	9.01
Average ($\bar{x}$)	1.41	2.21	2.57	7.08	3.62	9.65
D (1130 kg/ha fertiliser)						
12	1.55	3.35	2.38	7.00	4.90	9.38
46	1.12	5.13	2.80	7.39	6.25	10.19
34	1.89	3.71	1.76	6.87	5.60	8.63
23	1.69	5.32	2.96	8.00	7.01	10.96
Average ($\bar{x}$)	1.56	4.38	2.48	7.31	5.94	9.79

TABLE 7.1 (b)

Extractives in a 6-year old *E. globulus* tree from Treatment C (565 kg/ha) expressed as a of the oven-dry weight of samples*

Sample height (m)	Wood		Bark		Total	
	Ether	Methanol	Ether	Methanol	Wood	Bark
0.25	0.28	2.16	1.17	7.49	2.44	8.66
1	0.35	2.21	1.77	7.69	2.56	9.46
3	0.27	2.16	2.00	17.24	2.43	19.24
5	0.28	2.16	2.11	17.52	2.44	19.63
7	0.29	2.17	2.25	18.01	2.46	21.26

* The tree selected was one whose dbh was closest to the mean diameter of the treatment plot. Samples were extracted in the order listed.

Results in the brackets indicate non-significant subsets at P=0.05.

7.5 Analysis of results

The data relating to wood and bark in Table 7.1 (a) were subjected to the student Newman Keuls test (SNK) which is a multiple comparison of means based on equal sample sizes. Results of this analysis are shown in Table 7.2 (a) and (b).

Fertilisation had no apparent effect on the quantities of ether soluble extractives contained in the wood. However the amount of methanol soluble extractives in Group D, which had 1130 kg/ha of fertiliser, was significantly different at the 95% level of probability from the other two treatments (B and C) and the Control A.

This agrees with the findings of Erickson and Lambert (1958) in a study of the effects of fertilisation and thinning on the chemical composition of young Douglas-fir. They concluded that additional amounts of alcohol-benzene soluble extractives were associated primarily with fertilisation.

The amount of extractives in the bark of the sample trees was about the same for all groups. Fertilisation had no statistically significant effect. However there is a small progressive increase with increasing levels of fertilisation.

In the tree selected for height sampling the amount of ether soluble extractives in the wood was very similar at all levels. The quantity of methanol soluble extractives was highest, but not significantly so at the 1m level. The amounts of both ether and methanol soluble extractives were not significantly different whatever the height sampled.

TABLE 7.2 (a)

The mean amounts of extractives in the wood of 6-year old fertilised *E. globulus* expressed as a percentage of the oven dry weight of samples.

Treatment	Ether ($\bar{x}$)	Methanol ($\bar{x}$)
A (0.0 kg/ha fertiliser)	1.32	2.03
B (188 kg/ha fertiliser)	1.36	2.18
C (565 kg/ha fertiliser)	1.41	2.21
D (1130 kg/ha fertiliser)	1.56	4.38

The results within brackets are not significantly different at P=0.05.

TABLE 7.2 (b)

The mean amounts of extractives in the bark of 6-year old fertilised *E. globulus* expressed as a percentage of the oven dry weight of samples.

Treatment	Ether	Methanol
A (0.0 kg/ha fertiliser)	2.20	6.82
B (188 kg/ha fertiliser)	2.77	6.86
C (565 kg/ha fertiliser)	2.57	7.08
D (1130 kg/ha fertiliser)	2.48	7.31

Results within the brackets indicate non-significant results at P=0.05.

In the bark, there was a great increase in extractives from the 3m level; the highest being at the 7m level. The total amounts of ether and methanol soluble extractives at the 0.25 and 1m levels were almost the same and were significantly lower than those at levels 3, 5 and 7m at the 95% level of probability. The amount at the 7m level was the highest but not significantly different from that at the 3 and 5m levels. (Table 7.1(b)).

7.6 Effect of extractives on pulp and paper properties

7.6.1 Yield

During delignification in chemical pulping most extractives are rapidly dissolved. A small portion is, however, usually retained by the pulp produced. Consequently wood with normal lignin content but with a high extractives content usually gives lower yields of pulp on a weight basis. This is why some species with high amounts of extractives are not favoured by the pulp and paper industry. The discovery of an economic use for these extractives may help overcome this adverse effect, e.g. *Castanea dentata* (Chestnut) which yields tannins if the chips are extracted before pulping.

Several eucalypts that can be pulped satisfactorily have relatively low yields due to the high amounts of extractives they contain, e.g. the yield of unbleached soda pulp from mature jarrah (*E. marginata*) was 28%, from Karri (*E. diversicolor*) 38% and from mountain ash (*E. regnans*), with a low extractives content, 43% (Benjamin, 1923).

Kino veins or pockets not only increase alkali consumption but greatly reduce yield.

In this study, it was found, in the Kraft pulping of 6-year old *E. globulus* wood and wood plus bark, the total yield decreased from 56% for debarked stemwood to 53% for unbarked stemwood and even decreased further to 50% when unbarked branches were added to the furnish. The progressive decrease is probably due to the increasing extractives content of added portions to the debarked wood (Table 7.3(a)). Also in the NSSC "strong grades" the total pulp yield also decreased from 70% for debarked wood to 67% for unbarked wood (Table 7.3 (b)).

7.6.2 Chemical consumption

The suitability of wood for chemical pulping depends not only on the yield of pulp with desirable properties but also the quantity of chemicals consumed. The properties of the pulp itself depend on the amount of chemical used, time and temperature of cooking. Since time and temperature can be controlled at will, the amount of chemical consumed becomes important.

The inclusion of bark when Kraft pulping *E. globulus* increased the active alkali (Na_2O) consumed per a.d. tonne of pulp by about 19% but the yield still dropped from 56% to 53% (Table 7.3 (a)) because of the greater quantity of extractives in the bark. Cooking conditions were the same.

TABLE 7.3 (a)

Kraft pulping data of 6 year old *E. globulus* fertilised at 1130 kg/ha.* +

Property	Debarked Stemwood	Unbarked Stemwood	Unbarked Stemwood & Branches
Total yield %	56	53	50
Active alkali (% Na_2O)	13	15	16
Kg active alkali/air dry (a.d) tonne of pulp	210	250	290
M^3 wood/a.d. tonne of pulp	3.1	3.5	3.7
A.d tonne of pulp/hectare/year	2.7	3.2	3.6
kappa number	20	25	28
swelling ratio	90	80	70

* Only this group was pulped since a recent refined biomass determination has shown the highest fertiliser level to be the most effective in improving growth rate (Cromer, Raupach, Clark and Cameron, 1975). Earlier, attention had been focused on Group C (565 kg/ha of fertiliser) as this level of **fertilisation** was thought to produce maximum growth rate (Cromer and Hansen, 1972).

\+ Source: Farrington, Hansen and Nelson (1976). Utilisation of young plantation of *E. globulus*.

TABLE 7.3 (b)

NSSC pulping data of 6-year old *E. globulus* fertilised at 1130 kg/ha.*

Pulp Grade	Property	Debarked Stemwood	Unbarked Stemwood	Unbarked Stemwood & Branches
"Strong"	Total pulp yield (%)	70	67	-
	M^3 wood/air dry (a.d) tonne of pulp	2.5	2.8	-
	A.d. tonne of pulp/ hectare/year	3.4	4.1	-
	Kg of Na_2SO_3 consumed (a.d. tonne of pulp	156	179	-
	Kappa number	93	89	-
	Debris (Number $_2$ m^2) (0.2 mm^2)	98	130	-
"Corrugating"	Total pulp yield (%)	74	73	67
	M^3 wood/a.d. tonne of pulp	2.5	2.6	2.8
	A.d. tonne of pulp/ha/year	4.5	5.2	5.6
	Kappa number	114	116	115

* Source: Farrington, Hansen and Nelson (1976) Utilisation of young plantation of *E. globulus*.

The inclusion of branches further increased the alkali consumption by about 38%, from 210 to 290 kg of active alkali per a.d. tonne of pulp. Also extractives in the cell walls and lumina of some species also reduce the penetrability of cooking liquor especially the movement of liquid into the voids under hydrostatic pressure (Hillis, 1969). Cooking conditions, however, also affect the hydrostatic pressure.

7.6.3 Equipment corrosion and blunting of cutting tools

Problems such as corrosion of metals, blunting of tools and the adherence of insoluble coloured complexes to surfaces of mill equipment have been encountered since the pulping of eucalypts (ash eucalypts) started. Johnson (1947) and Perry (1952) reported various degree of corrosion of mild steel digesters used in Kraft pulping of ash eucalypts. This was thought to be due to the acidity of green eucalypt wood, the pH of which usually varies between about 4-6 but can be as low as 2.6 in some species. Little attention was given then to chemical reaction being responsible for the blunting of cutting tools.

Recent studies (McKenzie and Hillis, 1964) have confirmed the acidity effect but found that a number of polyphenols caused even more corrosion of metals and blunting of tools. They found that solutions of acetic acid (the main volatile acid in eucalypt wood) at a pH of 3.0 cause appreciable corrosion of carbon steel cutters within a short time. Also that weaker solutions of gallotannin and ellagic acid (an hydrolysis product of gallotannin) at higher pH attack iron more quickly than acetic acid. They are present in eucalypt wood in much higher concentrations than acetic acid.

Hillis (1971) found that extractives of these eucalypts used for pulping are characterised by the presence of ellagic acid, gallic acid and ellagitannins which are esters composed of ellagic (and sometimes gallic) acid and glucose. McKenzie and Hillis (1964) found a relationship between the number of vicinal hydroxyl groups present and the degree of corrosion by chelation.

Gardner and Hillis (1962) and Lees and Nelson (1967) found that under pulping conditions, the insoluble ellagic acid forms very insoluble, green-yellow complexes with inorganic ions. These coloured deposits collect and adhere strongly to many parts of the mill equipment lowering efficiency. Some of it has even been found to be entrained in the pulp itself increasing bleaching demand as mentioned in the next section. However deposits on equipment can be removed with sodium hypochlorite solutions at pH 8.5-10 or strong nitric acid (McKenzie, Pearson, Stephens, 1968).

The extent of corrosion was not measured in the pulping of the 6-year old *E. globulus*.

7.6.4 Colour and Bleachability

Investigations of substances responsible for loss in brightness of pulp and paper during manufacture and storage have been mainly centred around the carbohydrate and lignin portions of wood. Carbohydrates have been found to be responsible for a weak yellowing of pulp and paper on aging. Residual lignin in pulp has also been shown to be a source of colour when exposed to light (Claesson, Olson and Wennerblom 1968). On the other hand less consideration has been given to the influence of extractives since the amounts of these, in coniferous species most favoured for pulping are quite low. Now that hardwoods especially eucalypts containing a lot of extractives are also being used by the pulp and paper industry their effects on colour are gradually being investigated more.

Buttrick (1915) was the first to suggest that extractives may affect the colour of pulp when he ascribed the difficulty encountered in bleaching chestnut pulps to high tannin content. Hoge (1954) concluded that the yellow colour of sulphite pulp from Douglas fir was due to the presence of quercetin, a product of the sulphite oxidation of dihydroquercertin. Dean et al (1956) found the addition of black liquor in soda cooking of alpine ash (*Eucalyptus delegatensis*) resulted in an increased bleaching requirement which could not be explained in terms of lignin alone or reduced delignification. They found the time of contact of black liquor with the pulp during the pulping of alpine ash and other species was a big factor affecting bleach requirement. The increase in bleach requirement has been found to be due to the presence of alkali-oxidized ellagitannins in the black liquor which supplemented those produced by the extractives in the wood (Hillis, 1969).

Tardiff (1959) showed also that extractives are responsible for a considerable loss of brightness of eucalypt groundwood pulp and newsprint during manufacture and storage. Since no extractives are removed during groundwood preparation, the effect these have on colour is usually more pronounced than in other types of pulp and paper.

Bowman and Nelson (1965) also found a roughly linear relationship between brightness and the amount of methanol soluble extractives removed from *Eucalyptus capitellata* (Table 7.4). Table 7.5 shows however that the effect of removing methanol soluble extractives on brightness amongst eucalypts varies, the greatest effect from the species listed being on *E. sieberi*. They also found loss of brightness in groundwood from unbarked logs was more pronounced than that from debarked logs so that bleaching requirements increased.

Recent research on the effect on colour of different components of the polyphenolic extractives of alpine ash using chromatography by Hillis (1969) has shown that polyphenolic extractive and kino give with alkali more colour than lignin does and in addition, form insoluble material in some cases. Evidence now exists that combination between the alkali reaction products and cell wall does occur in the zone of increased UV (Ultra-Violet) absorption (due to either lignin or polyphenols or both) near the middle cell wall (Wardrop 1962). Hillis (1969) also found that under alkaline conditions, the colour formed by the same weight of materials is most intense with leucoanthocyanin polymers such as kinos,

TABLE 7.4

Effect of methanol extraction on the brightness of Kraft pulps from *E. capitellata*.*

	Number of Extractions (95% Methanol, 19°C, 6:1 V/W)			
	0	1	2	3
Time of Extraction	0	6hr	19hr	6days
% Extractives removed at each stage	0	4.95	1.21	1.82
Total Extractives removed %	0	4.95	6.16	7.98
Pulp yield	43.7	43.7	41.8	41.9
% Screenings loss	0.92	0.61	0.22	0.11
Brightness (G.E. Units)	18.5	22.4	23.9	26.7

*Source: Bowman, Nelson (1965). The effect of Extractives on the colour of Eucalypt pulps. Paper presented at Appita Conference 1965.

TABLE 7.5

Effect of methanol soluble extractives extraction on brightness of Kraft pulps from various species of Eucalypts.*

Sample	% Extractives Removed (E)	G.E. Brightness B	AB/AE
E. consideniana	0	10.4	0.8
	5.53	15.0	
E. Capitellata	0	19.2	1.0
	3.28	22.5	
E. Eugeniodes	0	18.5	1.5
	5.73	26.9	
E. Mulleriana	0	17.4	1.7
	6.42	28.3	
E. Obliqua	0	13.0	2.0
	4.97	23.1	
E. Cypellocarpa	0	14.5	2.0
	6.73	27.7	
E. Sieberi	0	15.6	2.5
	4.12	25.8	

* Source: Bowman and Nelson (1965). The effect of extractives on the colour of Eucalypt pulps. Paper presented at 19th Appita Conference, 1965.

strong with ellagitannins and weaker with gallic acid and catechin. The strongest colours were formed with Kraft pulping liquors and less with NSSC liquors. He concluded that ellagitannins are primarily responsible for the colours in the alkaline pulping liquors and not ellagic and gallic acids formed by alkaline hydrolysis.

When Kraft pulps from 6-year old *E. globulus* from Treatment D (1130 kg/ha of fertiliser) trees, were bleached, all samples gave 88% G.E. brightness. However, inclusion of bark and branches increased bleaching chemical consumption, even then bleached yields (expressed as oven-dry weight of pulp and wood) were lower from unbarked stemwood and branches 92% and 47% respectively compared with 95% and 53% for debarked stemwood alone (Table 7.6). Loss of brightness on exposure under controlled conditions was greater for pulps from wood and bark than from stemwood alone.

Tardiff (1959) investigating the loss of brightness over short periods of time found higher losses in pulp mixtures with a high proportion of eucalypt groundwood pulp and far less in mixtures with a high proportion of softwood pulp. This was due to the high amounts of ellagitannins in the mature eucalypt wood. Hillis (1962) found that the rapid discolouration of eucalypt groundwood was largely due to oxidative changes to the vicinal hydroxyl groups in polyphenolic extractives when exposed to air and sunlight. In his latest work, he found some of the variation and increase of colour in commercial NSSC eucalypt pulps may also be due to a decrease of pH during pulping owing to the high acidity and high polyphenol content of some woods and to other causes (Hillis, 1969). He also found that ease of bleachability attributable to conifer pulps and the difficulty encountered with pulps from mature eucalypt woods was due to the high amounts of polyphenolic extractive compounds with two or particularly three vicinal phenolic hydroxyls, the eucalypts contain. Also due to strong retention of oxidized leucoanthocyanins by cellulose material.

In Table 7.6, there is a sharp drop in brightness of pulps in which bark has been included. The high amount of polyphenolic extractives being responsible. After 18 hours at 105°C there was a drop of 10 G.E. units in the brightness of bleached pulp from stemwood plus bark and branches; for bleached pulp from stemwood alone a drop of 7 G.E. units in brightness was recorded.

7.6.5 Spent liquor recovery

The presence of large amounts of extractives have been responsible for the problems associated with chemical recovery in alkaline pulping eucalypts (Hillis, 1962). Hillis and Carle (1959) suggested that ellagic and gallic acids, components of the tannins in pale coloured eucalypts, may be responsible.

In Kraft pulping of 6-year old fertilised *E. globulus* only the potential burning properties of the spent liquor were investigated. The swelling ratio values of both stemwood and those with bark, suggested good burning properties. However, the inclusion of the bark reduced the swelling ratio

TABLE 7.6

Bleaching of *E. globulus* Kraft pulps from 6 year old fertilised trees using the sequence HC-E-D-H+

	Unbleached Kappa No.	Chemicals (% o.d. unbleached pulp) 1st Stage Cl_2	3rd Stage ClO_2	NaOH	(Yield (% o.d.) Unbleached Pulp	Bleached Pulp on o.d. pulp	on o.d wood	Brightness (% G.E.) 3rd Stage	4th Stage	1 hr at 105°C	18 hr at 105°C
Debarked Stemwood	19.8	4.65	0.5	0.05	55.8	94.6	52.7	89.4	90.6	86.7	83.5
Debarked or unbarked stemwood	18.9	4.3	0.4	0.04	55.0	94.9	52.2	85.9	89.1	85.9	81.6
Unbarked stemwood plus branches	27.3	5.8	0.5	0.05	50.8	91.6	46.5	85.9	88.0	83.9	78.7
Unbarked stemwood plus branches (screened)*	24.5	5.0	0.5	0.05	50.9	93.2	47.4	85.9	88.7	83.0	78.7
Whole tree	27.0	6.1	0.5	0.05	45.6	91.1	41.5	78.0	81.3		

* Fines (-4.8mm) were removed before Kraft pulping.

Bleaching Conditions

1st stage (HC) - 40% total Cl_2 as $Ca(OCL)_2$, 60% as Cl_2 water. Consistently 3%. Temperature 25°C.

2nd stage - 1.5% NaOH on o.d. pulp. Consistency 12%. Temperature 60°C.

3rd stage - Consistency 6%. Temperature 70°C.

4th stage - 0.25% $Ca(OCL)_2$ and 0.4% NaOH on o.d. pulp. Consistency 12%. Temperature 40°C.

\+ Source: Farrington, Hansen and Nelson (1976). Utilisation of Young Plantation of *E. globulus*.

of the black liquor (Table 7.3(a)).

When making handsheets from the Group D treatment trees of 6-year old *E. globulus*, constant sticking of handsheets to the plate of the handsheet making machine occurred. Whatever may be the cause, this could be due to the association of extractives in the pulp with the metal.

Finally, it is worth mentioning that the problems caused by extractives should be less in these young fast-grown eucalypt species due to the low amount of heartwood formed in the short rotation period. Already the Portuguese experience with *E. globulus*, with its rapid growth rate and short rotation age indicates that utilisation is possible before significant amounts of heartwood with its accompanying high extractives content are formed. Also according to Hillis (1969) thick walls impede the diffusion of pulping liquors and hinder removal of extractives while it is easier to do this with thin walled cells. Fertilisation significantly reduces double wall thickness in this study; this effect will help to offset the increased methanol extractives in the fertilised trees.

CHAPTER 8

THE EFFECT OF MEASURED ANATOMICAL CHARACTERISTICS ON THE PULP AND PAPER PROPERTIES OF FERTILISED AND UNFERTILISED SIX-YEAR OLD *E. GLOBULUS*.

8.1 Introduction

Among the factors which are related to the properties of the wood fibres and which are believed significant in determining the quantity and quality of the pulp which can be produced from different wood species are wood density, proportion of summerwood, width of annual ring, position of wood in the tree, age of the wood, fibre length, fibre diameter, ratio of fibre length and fibre diameter, fibre wall thickness and fibril angle (Besley, 1959). Of all the above listed characteristics, many of which are interdependent in any case, wood density (gms of moisture free wood per cubic metre of green wood) is the most important and useful single factor in wood quality determination for pulping purposes. This being due to its direct relationship to yield of fibre by any one pulping process and set of conditions and because of its association with certain paper strengths. According to Wangaard (1958) the importance of wood density in influencing pulp yield is brought out more clearly when yield is expressed on the basis of weight per unit volume of original wood and the near constancy of pulp yield per unit of dry weight of wood has reduced the practical problem of estimating yield per unit volume of wood to a consideration of the density of wood.

It is not surprising, therefore, that the studies of relationship between wood density and paper properties have assumed growing economic importance in countries such as Australia which are utilising hardwoods for pulping or exporting hardwood chips. The understanding of the effect of wood density and structure on paper properties is already contributing to wider utilisation of hitherto unused species either alone or as part of homogenous chip mixtures or as pulps with special properties for inclusion in tolerable proportions in paper making furnishes (Colley, 1973).

This relationship between wood density and paper properties has been worked out diagramatically by Higgins, de Yong, Balodis, Phillips and Colley (1973). In their work which involved eleven eucalypt species, covering the basic density range from 0.31 to 0.88 g/cm^3, they found that the properties of hardwoods expected to exert the main influence are ratio of fibre diameter to cell wall thickness, the extractives content and the distribution, and the diameter and frequency of vessel elements. They found also that within the eucalypts at least, the vessel volume does not seriously interfere with the relationship between fibre cross-sectional dimensions and wood density.

According to Besley (1959) hardwood vessels although representing very small amounts of dry matter due to their thin walls may possibly affect bonding strength of pulp but the extent of their influence has not been measured separately. Vessels may have an important effect on surface property of paper because of their shape which renders poor bonding with fibres and when present on the surface tend to cause "picking" during printing (Colley, 1975). He found this vessel picking tendency to be greatly influenced by vessel size and shape, their area as presented to the printing surface, the morphology of fibres associated with the vessels and their frequency (ratio of vessel and fibre volume) and the effect of beating on them. Not only did he find that more vessels were picked out as their size increased but the area of individual inked surface which is removed increased and markedly affected the subjective assessment of the quality of the print. Beating and the lowering of vessel/fibre volume ratio were found to lower the picking tendency. However in their recent work (Colley and Ward, 1976) they found that the lowering of this vessel/fibre volume ratio did not lower pick numbers in the same amount in all eucalypt species. They found that *E. deglupta* vessels had an inherently greater influence on picking tendency than those from mixed pulps. The reduction of the vessel/fibre volume ratio in the pulp of this species to 1:1200, which would in most commonly used eucalypt pulps eradicate vessel picking problems, resulted in the pick numbers of the same magnitude as mixed pulps with a higher ratio of 1:308. Work is still being done as to the differences observed in the behaviour *E. deglupta* vessels.

Nevertheless no morphological characteristic was found to correlate well with picking tendency in all the individual species they studied.

The importance of the amount of vessels and their influence on pulp and paper properties was not investigated in this present study.

Lange (1959) concluded that properties of the products made from wood are obviously more or less related to the morphology of the wood fibres. In an oversimplified manner, he described this dependence in terms of two factors, the shape of the fibre and the structure of the wall. Therefore the effects of wood density and fibre morphology are almost completely inseparable. An attempt will be made to relate the individual characteristics including fibre length to the pulp and paper properties where possible.

In this section of this study, therefore the effect of fertilisation on these important measured anatomical characteristics and how these subsequently affect the properties of the pulp and paper is investigated.

It is only for the determination of strength (burst, tear index and breaking length) and other wood properties that pulp and paper was made from 6-year old unfertilised *E. globulus* (Control A) and fertilised *E. globulus* (Group D--1130 kg/ha of fertiliser) for comparison. For other paper properties only the Group D trees were pulped since a recent refined biomass determination had shown the highest level of fertilisation to be the most effective (Cromer, Raupach, Clark and Cameron, 1975) and therefore the level of fertilisation which will be adopted for future plantation programmes.

Most of the pulping was done by staff of the A.P.M. Limited. Appita standards were used for all the evaluations except where otherwise indicated. Evaluation was carried out at the A.P.M. Limited Research Laboratories in Melbourne, Victoria.

8.2 Literature review

A large number of investigations have been made on the influence of anatomical and chemical characteristics of wood fibres on the pulp and paper properties. Dinwoodie, (1965) reviewed fully the progress that has been made in this area of research. There are many conflicting conclusions in literature with respect to the influence of anatomical characteristics on paper properties. Dinwoodie (1965) in his review tried to assign these conflicting results to faulty experimental design or biased methods of analysing the data or to misinterpretation of the results.

8.2.1 Burst and tensile strengths

Early investigations generally assumed that tensile strength (breaking length), burst strength and other strength-properties were primarily determined by fibre length. It is not surprising therefore that there has been little evidence to support this claim except that most raw materials used at that time were usually long fibred e.g. rags and cotton.

Cross and Bevan (1916) were the first to suggest that other factors were involved especially the number and nature of contacts between fibres. Later Klemm (1928) and Strachan (1925) suggested that the ratio of fibre length to fibre diameter should be considered rather than length by itself. It was not until the publication of the works of Benjamin (1923) and Nilsen (1926) that doubt was finally cast on the importance of fibre length in pulp strength properties.

Benjamin (1923) showed that short fibred Australian eucalypts gave satisfactory pulp strengths and Nilsen (1926) comparing pulps from spring wood and summer wood of pine found spring wood pulp had a higher breaking length. Nilsen's work led eventually to the postulation that breaking length and burst strength were inversely related to the percentage of summer wood. This was later extended to density since Hildebrandt (1962) suggested the use of density rather than the percentage of summer wood as the latterdid not always give a reliable indication of density.

Dadswell and Watson (1962) and Haywood (1950) found paper properties were dependent on the flexibility of fibres whether this was recorded as density effect as above or as an effect of cell wall thickness, cell wall area relative to total cross-sectional area (Mühlsteph ratio, Mühlsteph, 1941) or as the ratio of lumen diameter to fibre diameter (coefficient of flexibility). Petroff and Normand (1961) found a correlation coefficient of 83% between the coefficient of flexibility and breaking length. This ratio has since been used to classify trees of Zaire with respect to suitability for pulping by Istas, Heremans and Rackelboom (1954).

Dinwoodie (1965) however warned that most of the results should be accepted with caution since only one factor was allowed to vary at a time. The few investigations in which density and fibre length were both critically considered generally indicated that although the fibre length or ratio of fibre length to diameter (felting coefficient) is important (Pētēri, 1952, 1954; Wangaard 1962), the principal factor involved is a density term either density on its own or cell wall thickness (Jayme, 1958, Barefoot *et al*, 1964), or the ratio of twice the cell wall thickness to lumen diameter (Runkel, 1952).

Tamolang and Wangaard (1961) were among the first to employ multiple regression to determine quantitatively the various factors influencing pulp properties. In their study of 15 tropical and temperate hardwoods they accounted for 85% of the variation in burst strength and 87% of the variation in breaking length of unbeaten pulp in terms of lumen width directly and fibre diameter and specific gravity indirectly. Specific gravity was significant only at the 10% level. Wangaard (1962) in the extension of the above work by the addition of three more species, found unbeaten breaking length and burst to be significantly influenced by the ratios of lumen diameter to fibre diameter and fibre length to diameter both positively, the former variable being more important than the latter. Both variables accounted for 87% of the variation in breaking length and 83% of the variation in burst. In later work with 17 Philippine hardwoods, Artuz-siegel, Wangaard and Tamolang (1968) found that breaking length and burst of unbeaten pulp were dependant on cell wall thickness and specific gravity inversely. Also they also observed an indirect effect of the ratio of lumen diameter to fibre diameter on burst. Sheet density and specific gravity accounted for 89% of the total variation in burst and breaking length of unbeaten pulp.

The importance of fibre strength in sheet strength development was first demonstrated by Van den Akker *et al*

(1958) and this influence was later confirmed by Wangaard (1962); Kellogg and Wangaard (1964), Artuz-Siegel, Wangaard and Tamolang (1968). Artuz-Siegel *et al*. (1968) found the ratio of lumen diameter to fibre diameter and fibre strength were the main influencing variables on pulp beaten to 450 ml CSF (Canadian Standard Freeness), the influence of the felting coefficient was no longer significant. They observed the same trend as beating progressed, and explained the positive effect of lumen to fibre diameter ratio as a reflection of the influence of fibre to fibre bonding and effect of fibre strength being more enhanced in more compact sheet with high lumen to fibre diameter ratios. This confirmed the earlier work of Tamolang, Wangaard and Kellogg (1968) and of Kellogg and Wangaard (1964).

However, Barefoot *et al* (1964) found tracheid characteristics associated with wood density were most important in determining strength properties. In their studies they found that at least 93% of the variation of burst-ratio, tensile and tear strength could be accounted for in terms of tracheid fibre morphology. Matolcsy (1975) working on Kraft properties of Balsam fir (*Abies balsamea L.* (Mill)) reported that burst and breaking length were affected more by specific gravity, the only variable which was significantly correlated though negatively at 10% for beaten pulp at 500 ml CSF.

8.2.2. Tear index

A lot of uncertainty and some doubt still exists with respect to the most critical factors which contribute to tear strength of paper. There is no universally accepted set of variables which determine tear; on the contrary, perhaps the greatest variance in results is connected with this property. Directly opposite views are held by many workers on the influence of fibre length on tear. Many have derived a positive effect, but a few state that fibre length has no effect or if any, a negative one.

As with other strength properties tear was considered to be controlled by fibre length. Clark (1942) and Watson and Dadswell (1961) found tear was controlled by fibre length or weighted length of the fibre. However Pēteri (1954) and Petroff and Normand (1961) could not find any correlation between tear and fibre length but recorded a significant direct correlation with the ratio of fibre length to diameter.

Nelson *et al* (1961) in their comparative investigations of dicotyledons reported no relationship between tear and either fibre length or fibre length to diameter ratio. Barefoot *et al* (1964) accounted for 89% of the variation in tear by wood density while the inclusion of fibre length did not significantly increase the variation accounted for by the regression.

Watson *et al* (1952) found density or cell wall thickness to be the most important controlling influence on tear, even though they assigned some importance to fibre length. Annergen *et al* (1962) confirmed this in softwoods when they reported cell wall thickness to be the principal factor controlling tear while tracheid length was of secondary importance. Tamolang and Wangaard (1961) and

Wangaard (1962) found both fibre length and density or cell wall thickness to be almost of equal importance in their study of tropical and temperate hardwoods; fibre length was slightly more significant than cell wall thickness even though both variables were significant at the 1% level.

Dadswell and Watson (1962) Watson and Dadswell (1964) found fibre length to be most important factor in both softwoods and hardwood pulps. Cell wall thickness being more important in long fibred pulps and relatively unimportant in short fibre ones.

In a recent work by Artuz-Siegel *et al* (1968) on 17 Philippine hardwoods, they reported that the effect of fibre length on tear depended heavily on the beaten state of pulps. They found tear factor in unbeaten pulps was affected by the product of lumen to fibre diameter ratio and the reciprocal of specific gravity and also of fibre length to diameter ratio, the combined effect of them accounting for up to 78%. In beaten pulp (450 ml CSF), fibre length, fibre strength and Runkel ratio were the most important factors, all accounting for up to 83% of the total variation observed in tear strength. They found fibre length and fibre strength to be the predominant factors governing tear resistance and explained that this was due to their importance in determining the work involved in pulling out fibre from the sheet and in the case of well bonded fibres, in rupturing them. This has been confirmed by Tamolang, Wangaard and Kellogg (1968). As beating progressed to 300 ml CSF the effect of Runkel ratio decreases, since at this stage more fibres are ruptured in tear test due to increased bonding. They explained that the range of data has an important influence on the extent of variation accountable for by fibre length and the ratio of fibre length to fibre diameter. They found the wider the range the higher the fibre length influence. That the range of data has an effect on regression accountability has been pointed out by Dinwoodie (1965).

Wangaard and Williams (1970) found that fibre length is unimportant on tear resistance at low sheet density and also at some variable distance beyond the critical level of bonding, fibre length starts to have an adverse effect upon tear resistance. Also fibre strength has no effect on tear at sheet densities so low that tear occurs entirely through fibre pull out rather than rupture. It appears therefore the influence of the two variables are only clearly explainable when sheet density is considered.

Matolcsy (1975) found that tear at 500 ml CSF was dependent on fibre length, double wall thickness, the ratio of fibre length to fibre diameter and fibre diameter all positively in decreasing order of importance. In his multiple regression, only fibre length and double wall thickness were significantly correlated at the 5% level.

Jayme (1958) indicated that an optimum level of hemicellulose, contributes to tear strength of pulps while an increase in lignin content would result in a decrease.

Balodis (1963) critically examining the tear test itself concluded that it was only a compromise between strength and the effect of concentration of shear stresses. Dinwoodie (1965) believes also that the tear test usually employed, is a poor measure of strength of paper; concludes, that the principal properties influencing this empirical test in both beaten and unbeaten sheets are fibre length, cell wall thickness and probably also fibre strength.

8.2.3 Other paper properties

Fold, another paper characteristic, has received less attention than the above strength properties. Dinwoodie (1965) concluded that fibre flexibility and fibre length are the principal factors which influence folding endurance but the relative importance could not be determined due to scanty information.

Porosity according to Watson, Wardrop, Dadswell and Cohen (1952) appear to be related to density directly and indirectly to fibre flexibility (Murray and Thomas, 1961). Doughty (1932) noted that air permeability of sheets increase markedly with increase in fibre length.

There is a fairly general consensus that bulk of the paper is proportional to the density of the wood (Watson and Hodder, 1954) or to some property highly correlated with density such as the percentage of summer wood (Pillow, Chidester and Bray, 1941; Hammond and Billington,1949; Watson, Wardrop, Dadswell and Cohen, 1952) or cell wall thickness (Jayme, 1958) or the ratio of cell wall thickness to lumen diameter (Runkel, 1952). However Wangaard (1962) and Dadswell and Watson (1962) recorded a secondary effect of fibre length on bulk even though it is difficult to interpret the direct effect of fibre length on bulk.

The opacity of paper is correlated with the bulk since it measures the amount of interreflection of light between individual fibres so that the same factors which influence bulk also influence opacity (Masses, 1936). Harrison (1962) however found that beating which increases interfibre bonding reduces opacity.

8.3 Pulping of 6-year old fertilised *E. globulus*

The pulping was carried out at the Research Laboratories, A.P.M. Limited, Melbourne, Victoria. As there was limited capacity to evaluate the wood and pulp properties, this part of the study was confined to treatment D, the level of fertilisation that will be adopted. However the pulp data of the different treatments and the inclusion of bark and leaves were supplied by the staff of A.P.M. Limited.

8.3.1 Pulping materials

One tree was selected from the buffer zone of every plot in treatment D (the buffer zone refers to the two outer rows of each plot which were left as a buffer with 36 trees left in the centre as measured plots). Each tree selected had approximately the mean diameter calculated for the meaured plots. In those plots where diameters were not measured the mean height was used for selection (Cromer, 1971). The trees were felled, the branches cut off at their bases and the leaves removed. Diameter measurements were made over bark every 0.6 metre up the stem for volume table calculations. (Cromer, Hansen, 1972).

The stem was then cut at these points and 3.2cm lengths removed from each 0.61 metre lengths. The 3.2cm lengths and the leaves and branches were left for the biomass determination. The remaining 6.3cms lengths of stem were set aside for evaluation of wood and pulp properties.

To the above reserve, two other sample trees were again taken from the buffer zone of each plot. The trees selected were those with diameters approximating 1.6 standard deviations above and below the mean tree sampled previously so that on the whole about 95% of the population distribution was sampled. Extra sample trees were further removed from uncultivated plots of both treatments to obtain sufficient wood for pulp evaluation. About 24 trees were taken from treatment D.

The green stemwood was debarked and chipped in a laboratory chipper at the A.P.M. Research Laboratories, Fairfield, Victoria, retaining the + 4.8 mm - 28.6 mm fraction. Over size chips were further reduced with a small axe to - 28.6 mm. The sampling method for wood samples from three treatments (A, C and D) for evaluation and comparison of pulp strengths was the same (Farrington, Hansen and Nelson, 1976). They also further reduced the bark and branches without leaves from Group D trees with a small axe and passed through a Rover compost shredder, fines were also retained in the bark and branch samples.

8.3.2 Pulping methods

The wood chips were sampled on the floor, thoroughly mixing them in 8 different lots. A handful of each lot was then taken to make up the cook charges.

Three different methods were used in pulping the wood chips:

(a) Kraft pulping

Cook charges equivalent to 400 gms of ovendry chips were used in an electrically heated rotating stainless steel lined digester. The liquor to wood ratio of 3.5 ml/gm ovendry wood chips, 22% sulphidity. The cooking schedule was 1 - 3/4 hour to 162^{o}C and 1¼ hours at 162^{o}C. Two cooks were done varying the active alkali charge in 1% Na_20 steps to determine the level at which maximum screened pulp was obtained (14 and 15% active alkali). An extra cook using 13% active alkali and 21-20% sulphidity was also done by Farrington *et al* (1976). For cooks of unbarked stemwood plus branches, the digester charges consisted of each component in the same o.d. weight ratio as determined for the original trees by Cromer (1971). Cooking schedules were the same.

(b) NSSC'C' pulping.

Two high yielding soda cooks of NSSC'C' type were carried out. In one cook of this 'C' type 9.6% NaOH and 3.2% Na_2CO_3 were used as cooking chemicals instead of the usual Na_2SO_3. In the other of this type only 12% NaOH was used and no Na_2CO_3 at all. All chemicals are expressed on the basis of oven dry wood weight). Liquid to wood ratio was as in Kraft cook. The cooking schedule was 2 hours at 170°C and ½-1 hour at 170°C.

But in the pure NSSC cooks carried out by Farrington *et al* (1976), 15% Na_2SO_3 and 3.5% Na_2CO_3 were used as main cooking chemicals for the "strong" grades, with a cooking schedule of 2 hours at 175°C and 1, 2 or 3 hours at 175°C. For "corrugating" grades, 10% Na_2SO_3 together with 3% Na_2CO_3 was used and the cooking schedule being 2 hours to 170°C and 1 hour at 170°C.

(c) Pure Soda Pulping

Two cooks were carried out using NaOH as the only cooking chemical. 25% NaOH was used for one and 22% for the other. Sulphidity being also 22%, liquor to wood ratio was the same as in previous cooks. Cooking schedule was also the same used for Kraft pulping.

The cooked chips from all the above processes were disintegrated in a propeller disintegrator for 10 minutes. They were then defibred in a raffinator with water recirculation to retain as much fines as possible. The raffinator used was 203mm laboratory one, type VA, filled with 200 mm defibring plates equipped with flanges. Three successive passes through plate clearances of 0.4, 0.1 and 0.05 were carried out before screening. The pulps were further washed on a 0.25 mm Packer screen. The rejects were further refined with a plate clearance of 0.02 mm to reduce their amounts below 1% of the pulp.

8.4 Paper testing and determination of Kappa number

The physical properties were determined on handsheets. The method used in making the sheets is breifly described below:

(a) Handsheet making procedure

Four samples each weighing an equivalent of 24gm of oven dry pulp weight were taken from each of the air-dry pulps from the different cooks. Each sample was made up to 2 litres using distilled water and given 3000 revolutions in a British disintegrator. They were slushed through the handsheet machine and thickened into pads. Three sample pads from each pulp sample were torn up by hand into pieces and were made up to 821 gm each by the addition of warm distilled water at a temperature of 25$\pm$2^{o}C. At this stage the 3 samples were now beaten in Lampen mill at various revolutions depending on the type of pulp and CSF (Canadian Standard Freeness). The fourth sample was unbeaten but given an extra 300 revolutions, as the beaten samples in a disintegrator. The pulp samples were now made up to 8 litres still using distilled water (this was done until the CSF stage as an essential precaution). A mechanical stirrer was used to prepare a uniform pulp concentration in steel containers. The freeness was then determined for each sample and the temperature at which the determination was done recorded, usually this was between 22-25^{o}C. A second freeness was always done as a check. The pulp from this second freeness determination was then used in making a sheet for the determination of the stock concentration of the pulp suspension. The oven dry weight of the sheet was used in determining this. The freeness was later corrected at the measured temperature and stock concentration from a conversion table.

The pulp suspension was then made up to 16 litres using tap water at this stage. The initial sheet weight was determined from the oven dry weight of a sheet made from the 16 litre pulp suspension. This initial sheet weight with the stock concentration was used to calculate the required amount of dilution needed to give an approximately 60+2 g/m^{2}.

One set of 10 sheets each was made from each suspension, couched with 55.5 kPa force and then pressed dry in the sheet press. Finally they were placed on round steel plates and then in metal rings to prevent warping during drying in a conditioned room.

For the high yield soda cook type NSSC'C' in which carbonate was included as cooking chemical, two double weight sheets were made for the crush or Concora test.

(b) Testing

The physical properties of the dried sheets were tested in a pre-conditioned room where the sheets were

dried. The Appita standard method P208m-64 was used throughout the testing.
For tearing resistance (4 plies) 4 tests were carried out per set of handsheets. And for tensile strength, stretch and bursting 8 tests each and for air resistance 4 tests were done.

(c) Determination of Kappa number

This is essentially the number of mls of 0.1N potassium permanganate solution which would be consumed by 1 gm of moisture free pulp, and the results corrected to 50% consumption of the permanganate. It gives the relative hardness, bleachability or the degree of delignification of pulps.
A sample of air dry pulp weighing about 4gms was taken from each pulp sample. This was well torn up into pieces and made up to 2 litres using distilled water. The pulp suspension was given 300 counter revolutions in a disintegrator, and later the slush was filtered on a Buchner funnel, and the filtrate returned twice through the pad. This is to avoid loss of fines. The pad was air dried, torn into pieces again and left in an airconditioned room for 20 minutes.
Two samples were then weighted from it. A small sample about 0.001 gm or an amount that would approximately consume 50% of the potassium permanganate. The large sample about 1 gm or more was used to determine the moisture content of the air dry pulp. The small sample was disintegrated in 500 ml

of distilled water making sure that many fibres are not extensively cut. This sample was transferred to a reaction vessel, thoroughly rinsing the container with distilled water. The reaction vessel was put on a bath at near constant temperature of $25^{o}C \pm 0.1^{o}C$. A constant check was kept on this temperature. A vortex of 2.5cm in the solution was obtained using a mechanical stirrer.

A mixture of 100 ml of 4 N. sulphuric acid and 100 ml of 0.1N potassium permanganate solution was made and the temperature brought also to $25^{o}C$. This was added quickly to the pulp suspension. After 10 minutes, 20 mls of 1.0Molar solution of potassium iodide was added. This was then titrated with 0.2N sodium thiosulphate solution, with a few drops of 0.2% starch solution as an indicator being added to get a clear end point. A blank titration using same procedure was carried with no pulp.

Kappa Number was then calculated using the formula:

$$K = \frac{P.F}{W} \text{ where}$$

$$P = \frac{(b-c)n}{0.1}$$

b = ml of thiosulphate solution consumed in the blank

a = ml of thiosulphate solution consumed in the titration itself

n = normality of thiosulphate solution

f = factor correction to 50% permanganate solution (table for this available in the lab)

W = weight of sample in gms of O.D. pulp

K = Kappa number.

8.5 Results

The results are presented in three groups for this investigation. Tables 8.1 and 2 dealing with wood and Kraft pulp properties of three treatments of the four levels of fertiliser experiment (Groups A, C and D with 0.0, 565 and 1130 kg/ha of fertiliser respectively) are provided by Farrington *et al* (1976). Tables 8.3 and 8.4 (a), (b) and (c) are pulping data of the debarked stemwood only of Group D treatment by different pulping methods and conditions. Table 8.4 (a), (b) and (c) have been presented in their form to show effect of beating on some of the pulp properties. Lastly Kraft and NSSC pulping data of debarked stemwood, unbarked stemwood and unbarked stemwood with branches and leaves for Group D treatment are also presented in Table 8.5 and 8.6 (a) and (b) (Farrington, *et al* 1976). The data in these last two tables were interpolated from curves of handsheet properties against beating revolutions at 200 CSF for Kraft and NSSC "strong" grades and at 300 CSF for NSSC "corrugating" grades (Table 8.6 (b)).

Where possible the wood and pulping data for the 2 and 4 year old *E. globulus* have been included to show the trend of these properties over the years. These were done by Cromer and Hansen (1972).

Table 8.7 shows the influence of some determined arithmetic ratios on the strength properties of Kraft pulp of fertilised and unfertilised *E. globulus*. A summary of the statistics of linear regression Y=a+bx of some anatomical wood characteristics, that were determined in this study against pulp properties is presented in Table 8.8.

8.6. Discussion

8.6.1 Comparison of wood and pulp properties of fertilised and unfertilised *E. globulus*.

(a) Wood properties

The volume of wood produced by the fertilised groups was significantly higher than the control group. At age 6, the highest level of fertilisation treatment (Group D) was producing four times more wood than the Control (Table 8.2). However wood produced by the fertilised groups was found to be slightly but not significantly, lower in basic density (Table 3.2, p. 31). In Table 8.2, it can be observed that the mean annual increment of stem-wood is increasing with age; the rate of increase between ages 4 and 6 years being slower than between ages 2 and 4 years for both fertilised and unfertilised groups. The rate of increase has slowed down in the fertilised groups. The total dry weight of wood produced by treatment C at age two was found to be ten times more than Group A (Cromer and Hansen, 1972). It appears that effect of fertilisation has started to dwindle as shown in the rate of increase at age 6 years.

The lignin, pentosan and boiling water extractives content all decreased between the ages 2 and 4 years. At age 6, the extractive content had decreased below that in 4 year old trees, but the lignin and pentosan contents showed little or no further change. The lignin content of the control was only slightly (2%) higher than that of fertilised groups C and D, but not significantly so. The level of fertilisation did not have any effect on the amounts of these substances. As shown, these were the same in Group C (565 kg/ha) and Group D (1130 kg/ha). The boiling water extractives even though increased progressively with increasing amount of fertilisers, still the highest level of fertilisation was not significantly different from the other levels.

(b) Pulp properties

The data of Kraft pulp strengths as shown in Table 8.1 reflect no significant differences between the three treatments. Nevertheless, the fertilised groups produce pulps of slightly higher burst and tear index compared with the control.

The lower burst strength of pulp of the control may be explained in terms of the higher basic density and the morphological difference of its fibres compared with the fertilised groups. The trees of the control have thick walled fibres (significantly thicker than those of Group D, the highest fertilisation level) which do not collapse easily on pulping, are stiff, tend to retain their rounded or tubular shape during sheet formation and do not easily bond. The fibres of Group D, on the other hand have thin walls, collapse to flattened ribbons during pulping and have a higher fibre to fibre bonding resulting in pulp of high burst strength.

This is in agreement with the findings of Swartz and Bray (1941), Besley (1959), Dadswell (1960), Barefoot, *et al.* (1964) and Matolcsy (1975). It was found that the higher the ratio of lumen diameter to fibre diameter (coefficient of flexibility), the higher the burst strength of the pulp. Group D trees with the highest ratio also produced pulps of higher burst (Table 8.7). This agrees with the results of Gleeson (1966) who in his pulping studies with eucalypts, found those with a higher ratio than 0.45, produced high pulp strengths. Barbadillo (1967) has also confirmed this in the experimental pulping of some Spanish eucalypts. The importance of this ratio has also been shown by Artuz-Siegel, Wangaard and Tamolang (1968) in their studies with 17 species of Philippine hardwoods. The two princial factors they found, influencing burst and tensile strength of beaten pulps being coefficient of flexibility and fibre strength.

The ultimate effect of this ratio is enhancing fibre to fibre bonding, thus making more compact sheets with high strengths.

The variation of breaking length with fertilisation was not clearly defined. The Control and Group D teatments produced pulps of the same tensile strengths while the in-between Group C had a higher tensile compared to the two treatments above.

The explanation of the lower tear index of the pulp of Control trees can not be in terms of fibre length alone. Control trees have slightly but not significantly longer fibre lengths than the fertilised groups, but still had lower tear index. This is contrary to the traditional belief that this property is controlled directly by fibre length; evidence to support this belief have been put by workers such as Clark (1942) and Watson and Dadswell (1961). The variation of this property will be discussed more in the next section.

8.6.2 Association of measured anatomical characteristics with strength properties of Kraft pulp and 6-year old *E. globulus* (pulps were beaten to 300 CSF)

As pointed out earlier, past investigations on the influence of various anatomical characteristics and their relative importance, have produced a lot of contradictory results. In this study, like some past investigations, one factor was allowed to vary at one time in the linear regression of these characteristics with pulp properties. There is no doubt however that factors other than the varying one may vary concurrently. This has been pointed out by Dinwoodie (1965) in his review.

(a) Burst index

Of all the measured anatomical characteristics regressed against burst index only Runkel ratio (2W/L), felting coefficient (FL/D) and coefficient of flexibility (L/D) were significantly correlated with the property at the 5% level of probability, positively only with the last anatomical variable (Table 8.8). The three variables accounted for up to 99% each, for the variability in this property independently. The seemingly high correlations of these variables and of other variables that were not significant is due to the small number of observations.

(b) Breaking length

Fibre length was the only variable that was significantly correlated with breaking length at the 5% level of probability though negatively.

(c) Tear index

The influence of anatomical variables on tear strength is still the most uncertain and consequently more contradictions are found in literature. In the regression analysis only lumen diameter was positively and significantly correlated with this property accounting for up to 96% alone for the variations observed in this study. Strangely enough fibre length was very poorly correlated with this property (Table 8.8). The trend seem to indicate (Table 8.7) that the lower the Runkel ratio the higher the tear factor. In general this agrees with Runkel (1952) in his studies with tropical hardwoods when he found wood with lower Runkel ratio to be associated with high pulp lengths. Also Wangaard (1962) recorded this effect of Runkel ratio on tear resistance of three hardwood species but he adds that such fibres have to be exceptionally thick walled so that fibre to fibre bonding will be weak. The effect of high Runkel ratio is a resistance to collapse during beating and the associated opportunity for fibre to fibre bonding. This is consistent with the theory in which tear resistance is explained in terms of the amount of work involved in pulling out fibres of an entanglement in the sheet and, in the case of well bonded fibres, in rupturing the fibres themselves (Van den Akker, 1960). So that Runkel ratio, in facilitating improved bonding (when not too high) will also be effective in improving tearing strength only up to a point where a substantial number of fibres are ruptured when the sheet is torn.

In a fairly recent work by Tamolang et al (1968), they found the range of fibre length data has an important influence on the amount of variability accountable for by fibre length in tear strength. They found, the higher the range, the higher the influence. Dinwoodie (1965) also emphasised the effect of data range on regression accountability. The influence of fibre length in this study on tear resistance may have been masked by the smallness of the fibre length data range (0.87-0.95 mm). The mean fibre lengths of the different treatments were not significantly different.

Also Watson and Dadswell (1964) were able to explain the difference in tear resistance of *E. deglupta* and *E. moluccana* by the Runkel ratios of the two species. Both species have the same fibre length but with Runkel ratios of 0.37 and 1.94 respectively (Table 8.9). This resulted in a big difference in their tear index.

It thus appears that once a minimum fibre length was available, other anatomical variables assumed a higher influence on strength properties of pulp such as tear index. It should be mentioned that the higher but non-significant lignin content of the unfertilised trees may also contribute to decrease their tear strength which agrees with the work of Jayme (1958). The effect of lignin is to decrease interfibre bonding. Finally the differences in tear index as well as the other strength properties of pulps from the three treatments were only slightly varied.

8.6.3 Effect of different pulping processes, cooking condition, amount of chemicals and beating on pulp properties of debarked fertilised stemwood of *E. globulus* (1130 kg/ha)

(a) Yield

The NSSC "C" soda type cook produced higher pulp yields compared with all other processes. However the non-use of sodium carbonate in the same process type resulted in a slightly lower yield. In the soda cook, the increase in cooking chemical resulted also in a lower yield. The increase of the active alkali percent in the Kraft process from 14 to 15% did not affect the yield because the higher chemical percentage gave only a slightly higher yield of pulp (Table 8.3).

(b) Pulp Properties

The brightness of Kraft pulps was higher than for all other pulp types, the darkest pulp being those of NSSC "C" type cook (Table 8.3). The NSSC 'C' type cooks produced pulps of the higher Kappa Number than soda and kraft pulps which were not different. The Kappa Number decreased with increasing amount of cooking chemicals (Table 8.3).

The initial burst, tear and tensile strengths of Kraft pulps were higher than those of soda and NSSC 'C' type cooks and also responded to beating by rapid increases in these strength properties. The strength properties of NSSC 'C' type cook were the poorest and so was the response to beating (Table 8.4 (a, b and c)). The poor response to beating of soda and NSSC 'C' pulps may be due to the alkali used as the main cooking chemical resulting in a more complete removal of easy beating hemicellulose and related materials. On the other hand, the presence of sulphide in Kraft pulping chemical increased rate of delignification and helped to diminish the injurious effect of alkali alone on cellulose and hemicellulose materials. This goes to explain the better beating response of these pulps also.

8.6.4 Kraft and NSSC pulp properties of debarked stemwood, unbarked stemwood and unbarked stemwood with branches and leaves of *E. globulus* fertilised at 1130 kg/ha.

This section was included due to the increased importance of the fertilisation level, Group D, of the fertiliser experiment. Early indications were that the Group C (565 kg/ha level of fertilisation produced the maximum growth rate (Cromer and Hansen, 1972) and hence samples for this level of application were used to determine the wood and pulp properties of the two year old trees. Subsequent results for the 4 year old trees however showed the highest level of fertilisation (1130 kg/ha) to be more effective due to the refined biomass determination of Cromer, Raupach, Clarke and Cameron (1975). Hence this level of fertilisation which will be adopted had to be investigated as to yields, pulp properties, chemical consumption, etc.

(a) Kraft Pulps

(i) Yield and wood consumption

The pulp yield based on oven drywood was highest for stemwood alone and the inclusion of bark or branches with the stemwood reduced the yields. The pulp yield as tonnes per hectare per year was lowest for debarked stemwood. The inclusion of other components such as bark and branches significantly increased the pulp yield (tonnes/hectare/year) (Table 8.5). The inclusion branches increased yield in the above terms by about 33% and this steadily rose to 40% with the whole tree. Both yields in terms oven dry wood or tonnes per hectare per year increased with increasing age. (Data for two year old trees supplied by Cromer and Hansen, 1972).

The wood consumption expressed in cubic metre per air dry tonne pulp decreased with age. This is probably due to the combined influence of increasing pulp yield and beyond 4 years, increasing wood density. The inclusion of other tree components to the stemwood, increased wood consumption, the highest increase being ascribed to the whole tree pulping. The use of whole tree chips also considerably reduced the effective digester capacity.

(ii) Pulp properties

The pulp properties for debarked wood, unbarked wood and branches were very similar, although the inclusion of bark and branches resulted in increase consumption of cooking chemicals accompanied by small reductions in strength properties. At age 6 the tear index is still slowly increasing while the tensile and burst strengths appear to have reached their maximum. The increase of tear index probably reflects the importance of fibre strength or maturity as referred to earlier in the comparison of unfertilised wood pulps.

(b) <u>NSSC Pulps</u>

As for the Kraft, the pulp yield per hectare per year increased with increasing age especially between ages 2 and 4 years.

The inclusion of bark lowered yield, based on oven dry wood for a given Kappa Number but still increased pulp yield per hectare per year considerably (Table 8.6 (a)). The inclusion of bark or branches also increased chemical consumption, but had little effect on strengths except in the case of burst index of 2-year old samples. However in the corrugating grade pulps, the inclusion of branches significantly decreased the tear burst and concora (crush strengths (Table 8.6 (b)).

The swelling ratios for all samples were quite close though the addition of branches and leaves in the corrugating grades significantly reduced the swelling ratio of such black liquor. High swelling ratio indicates good burning properties of black liquor.

8.6.5 Optimum age of utilisation

The determination of the age of utilisation involves the consideration of many factors, the relative importance of which could vary from mill to mill and even for different end products in the same mill. As data available for plantation trees of *E. globulus* do not yet extend beyond 6 years, results from studies on regrowth natural forests in both Tasmania and Victoria have been included. These studies were carried out by the A.P.M. Limited Research Division. The conclusions that can be drawn from these many not be very accurate as this procedure adopted may not be strictly valid. However they are good indications of the trends until older plantation are available for study.

The important factors examined in these studies were wood (m^3) and active alkali (kg) consumed per a.d. tonne of pulp and burst index all of Kraft pulps (Fig. 8.1). The variation of these factors with age are for stemwood only. The data provided by A.P.M. Limited Research Division showed that burst index rises sharply over the first few years and probably reaches its maximum before or at 10 years of age. It then starts to decline between 40-50 years and after then only slightly. The presence of brittle heart and rot and the subsequent increase in the amount of broken fibres with increasing age could further result in a decrease in this strength property.

The alkali consumption also appears to have reached its minimum value around 10-20 years and slowly increases after this age. This could be due to the higher amount of heartwood formed and its accompanying higher amounts of extractives.

The wood consumption is continuously decreasing steadily with age due to increasing basic density of the wood and higher pulp yields.

Interpolating from the graph (Fig. 8.1), it thus appears that the optimum utilisation age for Kraft pulping will be around 10 years. At this age, burst index is maximum, chemical consumption is at its lowest value, and wood consumption has at least decreased considerably from the high amounts characteristic of the very young wood. Even then the use of older wood, (more than 10 years) will mean lower burst strength and increased alkali consumption.

The ultimate decision with respect to complete optimisation will have to depend on the consideration of other factors such as the strength values needed in the pulp for the product intended, the importance attached to the variables that have been considered and most importantly the cost of wood procurement which will take into account costs involving the land, plantation establishment and maintenance and harvesting. The fact that a certain volume of fertilised wood represents a lower value to a mill than does the same amount of wood from an unfertilised forest also introduces a complexity in all these considerations.

8.6.6 Conclusion

Fertilisation produced a significant increase in volume of wood yield per hectare compared with the Control Group (Table 8.2). The pulp strengths of the fertilised wood were higher though not significantly with the exception of the breaking length whose variation pattern was not clear. While the Control A had a higher tensile strength compared with the highest level of fertilisation, Group D, Group C fertilised at 565 kg/ha had the highest (Table 8.1) tensile value.

The inclusion of bark and branches resulted in the increased yields of Kraft and NSSC pulp per hectare accompanied with increased chemical consumption. The pulp strengths were not significantly lowered except in the NSSC corrugating grade pulps where the inclusion of branches affected burst, tear and crush detrimentally (Table 8.6 (b)).

The principal factors found to exert the main controlling influence on the pulp properties were the ratios of lumen to fibre diameter, fibre length to fibre diameter and Runkel ratio on burst index; fibre length on breaking length and lumen diameter on tear index (Table 8.8).

The importance of fibre length in tear strength development could not be revealed in this study. It appears therefore that within a narrow range of fibre lengths, the importance of fibre length in this respect becomes masked while other anatomical variables become more important. Fibre length is therefore only important in so far as a minimum length is required for bonding and fibre wall structure controls the optimum strength obtainable. It is worth remembering that the mechanism of tear is not still well understood. According to Dinwoodie (1965) tear strength in itself is a poor measure of strength of paper. Balodis (1963) even concluded that the test was a compromise between strength and the effect of concentration of shear stresses.

From the variation studies of burst index, m^3 wood and kg active alkali consumed per a.d. of Kraft pulp with age for old regrowth natural forest of *E. globulus* in both Tasmania and Victoria (Fig. 8.1), (A.P.M. Limited, 1976) it might be safe to say that a rotation age of 10 years will be adequate for plantation grown material. At this age the values for these variables are at their optimum. However other considerations especially cost still need to be taken into account for maximum optimisation decision to be made.

TABLE 8.1

Comparison of Stemwood Kraft* Strength Properties with Fertilisation for *E. globulus*

Property	Age (years)	Fertilisation Levels A(0.0 kg/ha)	C(565 kg/ha)	D(1130 kg/ha)
Burst index	2	7.2	8.0	-
	4	8.6	8.9	8.9
	6	8.5	8.6	8.7
Tear index	2	8.7	8.2	-
	4	10.5	10.0	9.8
	6	10.1	10.3	10.8
Breaking length (km)	2	9.9	11.0	-
	4	11.0	11.2	12.1
	6	12.2	12.6	12.1

* Source: Farrington, Hansen and Nelson (1976): Utilisation of Young Plantation of *E. globulus*.

TABLE 8.2

Wood Yield and Properties of Stemwood from *E. globulus* Fertilised at Four Different Levels at Different Ages*

Property	Age (years)	Fertilisation Treatment A(0.0 kg/ha)	C(565 kg/ha)	D(1130 kg/ha)
Mean annual increment (M^3 ha/yr)	2	0.28	2.63	-
	4	1.66	6.02	8.18
	6	2.12	6.27	8.40
Boiling water extract (%)	2	5.7	6.8	-
	4	6.0	5.2	5.2
	6	3.1	3.5	4.0
N/10 NaOH extract (%)	2	24	25	-
	4	21	21	20
	6	18	19	17
Lignin (%)	2	20	20	-
	4	17	17	17
	6	19	17	17
Pentosan (%)	2	24	24	-
	4	22	21	21
	6	22	22	21

* SOURCE: Farrington, Hansen and Nelson (1976) Utilisation of Young Plantation of *E. globulus*.

TABLE 8.3

The Yield, Brightness and Kappa Number of Debarked Stemwood Pulp of *E. globulus* Fertilised at the Rate of 1130 kg/ha. Under Different Pulping Conditions

	NSSC'C' (high yielding soda type cook)		Soda cook		Kraft cook	
Cooking schedule	2 hours to 170°C and ½-1 hour at 170°C		1 3/4 hours to 162°C and 1¼ hours at 162°C		Same as soda	
Main chemicals	9.6% NaOH and 3.2% Na_2CO_3	12% NaOH	22% NaOH	25% NaOH	14% Na_2O and 22% Sulphidity	15% Na_2O and 22% Sulphidity
Yield (%)	75.4	71.4	54.2	50.6	53.1	54.5
Brightness % G.E.	16.6	17.1	34.5	40.4	42.2	42.2
Kappa Number	103	89	20	19	21	19

TABLE 8.4 (a)

The Effect of Beating on Pulp Properties of *E. globulus* from NSSC'C' Soda Type Cook

Main Cooking Chemicals	9.6% NaOH AND 3.2% Na_2CO_3				12% NaOH			
Beat Revs	0	5000	10000	25000	0	5000	10000	15000
Free CSF	635	400	300	150	625	410	340	215
Tear Index	6.0	6.5	7.1	6.5	6.9	8.2	8.2	8.7
Break LN KM	2.71	5.31	6.30	8.23	3.37	6.53	7.52	8.40
Stretch PC	.9	2.0	2.1	2.9	1.3	2.2	2.5	3.0
Burst Index	1.25	2.71	3.25	5.22	1.72	3.30	4.37	4.70
Bulk CC/G	2.23	1.84	1.78	1.59	2.02	1.72	1.64	1.61
Air Res	.9	4.6	9.5	71.0	1.2	5.6	13.5	29.5
Gramm GSM	58.7	62.3	56.5	58.3	59.0	60.3	60.0	59.8
Crush N	157	252	286	390				

TABLE 8.4 (b)

The Effect of Beating on Pulp Properties of *E. globulus* from Soda Cook

Main Cooking Chemicals	22% NaOH				25% NaOH			
Beat Revs	0	5000	10000	20000	0	1000	20000	35000
Free CSF	630	485	430	320	600	550	285	225
Tear Index	6.4	10.0	10.5	10.9	5.2	6.7	9.5	9.5
Break LN KM	3.72	7.24	8.12	9.20	3.58	4.23	8.94	9.32
Stretch PC	1.5	2.6	3.0	3.5	1.4	1.7	3.2	3.5
Burst Index	1.66	4.31	4.68	5.81	1.83	2.09	5.55	5.84
Bulk CC/G	1.89	1.62	1.62	1.52	1.84	1.77	1.38	1.38
Air Res	2.2	5.0	5.6	13.1	1.8	2.3	11.2	26.5
Gramm GSM	64.4	59.7	60.5	59.6	58.7	58.7	58.2	60.1

TABLE 8.4 (c)

The Effect of Beating on Kraft Pulp Properties of *E. globulus*

ACTIVE ALKALI %	14				15			
Beat Revs	0	2500	6250	12500	0	5000	10000	15000
Free CSF	385	355	280	210	435	305	265	225
Tear Index	8.5	10.0	10.2	10.5	8.3	10.2	10.2	10.3
Break LN KM	6.51	9.81	11.04	11.84	5.57	10.50	10.47	11.28
Stretch PC	2.4	3.2	3.8	4.4	2.1	3.8	3.8	4.0
Burst Index	3.55	6.36	7.53	8.55	3.05	6.58	7.47	7.58
Bulk CC/G	1.59	1.46	1.40	1.37	1.63	1.44	1.39	1.30
Air Res	7.2	19.0	37.4	114.4	5.9	21.4	47.2	64.9
Gramm GSM	59.4	59.6	59.8	59.9	60.1	59.6	62.9	58.4

TABLE 8.5

Kraft pulping data for *E. globulus* fertilised at 1130 kg/ha at different ages (pulping was to maximum screened yield).*

Property	Age (Years)	Stemwood (Debarked)	Stemwood (Unbarked)	Unbarked Stemwood and Branches	Whole Tree
Active alkali (% Na_2O)	2	(14)	(17)	(18)	-
	4	13	16	16	-
	6	13	15	16	17
Total Yield (%)	2	(50)	(46)	(42)	-
	4	53	50	48	-
	6	56	53	50	46
Kg active alkali/o.d. tonne pulp	2	(250)	(330)	(380)	-
	4	220	290	300	-
	6	210	250	290	330
M^3 wood/o.d. tonne pulp	2	(3.5)	(3.9)	4.4	-
	4	3.4	3.8	3.8	-
	6	3.1	3.5	3.7	4.1
Tonnes o.d. pulp/ha/year	2	0.8	0.9	1.1	-
	4	2.4	2.7	3.4	-
	6	2.7	3.2	3.6	3.8
Kappa Number	2	(18)	(23)	(26)	-
	4	16	21	25	-
	6	20	25	28	30
Burst Index	2	(8.0)	(6.9)	(6.9)	-
	4	8.9	7.6	7.6	-
	6	8.7	8.0	7.7	7.4
Tear Index	2	(8.2)	(8.4)	7.9	-
	4	9.8	9.6	9.3	-
	6	10.8	9.9	9.6	9.1
Swelling ratio	6	90	80	70	50
Breaking length (km)	2	(11.0)	(9.7)	9.8	-
	4	12.1	10.8	10.8	-
	6	12.1	10.8	10.5	10.3

Data in brackets refers to Group C trees (565 kg/ha of fertiliser).

* Source: Farrington, Hansen and Nelson (1976) Utilisation of Young Plantation *E. globulus*.

TABLE 8.6 (a)

NSSC pulping data for *E. globulus* fertilised at 1130 kg/ha - strong grades. (Data in brackers refer to *E. globulus* fertilised at 565 kg/ha).*

Property	Age (Years)	Stemwood (Debarked)		Unbarked Stemwood	
Total pulp yield (%)	2	(67)	(65)	(64)	(62)
	4	72	67	66	63
	6	72	68	68	66
M^3 wood/o.d. tonne pulp	2	(2.6)	(2.7)	(2.9)	(2.9)
	4	2.5	2.7	2.9	3.0
	6	2.4	2.6	2.7	2.8
o.d. tonne pulp/ha/year	2	(1.2)	(1.2)	(1.4)	(1.4)
	4	3.2	3.0	3.6	3.5
	6	3.5	3.2	4.1	4.0
Kg Na_2SO_3 Consumed/o.d. tonne pulp	2	(175)	(198)	(185)	(205)
	4	137	182	171	204
	6	133	179	162	189
Kappa Number	2	(100)	(91)	(94)	(89)
	4	105	86	99	90
	6	101	85	94	84
Burst index	2	(4.4)	(4.6)	(4.4)	(3.6)
	4	6.1	6.9	6.4	6.7
	6	6.0	7.0	6.3	6.7
Tear index	2	(6.5)	(6.8)	(6.5)	(6.7)
	4	8.2	8.8	8.4	9.0
	6	9.6	9.2	8.1	9.1
Breaking length (km)	2	(7.4)	(7.5)	(6.9)	(6.3)
	4	9.6	10.3	9.8	10.2
	6	9.6	10.8	9.2	10.0
Debris count (No/M^2) ($o.2mm^2$)	2	(3950)	(3750)	(4000)	(2450)
	4	105	175	215	105
	6	145	50	-	130

* Source: Farrington, Hansen and Nelson (1976): Utilisation of young plantation *E. globulus*.

TABLE 8.6 (b)

NSSC pulping data for *E. globulus* fertilised at 1130 kg/ha (data in brackets refer to *E. globulus* fertilised at 565 kg/ha). All corrugating grades.*

Property	Age (Years)	Unbarked Stemwood	Unbarked Stemwood and Branches	Whole Tree
Total pulp yield (%)	2	-	(64)	-
	4	72	69	-
	6	74	73	67
M^3 wood/o.d tonne pulp	2	-	(2.9)	-
	4	2.6	2.7	-
	6	2.5	2.6	2.8
O.D. tonne pulp/ha/year	2	-	(2.0)	-
	4	3.9	4.8	-
	6	4.5	5.2	5.6
Kg Na_2SO_3 consumed/ o.d. tonne pulp	2	-	(128)	-
	4	99	111	-
	6	94	98	109
Kappa Number	2	-	(118)	-
	4	129	114	-
	6	114	116	115
Burst index	2	-	(1.9)	-
	4	4.1	3.8	-
	6	4.9	3.6	3.4
Tear index	2	-	(5.4)	-
	4	7.3	7.0	-
	6	7.7	7.0	6.8
Breaking length (km)	2	-	(3.9)	-
	4	7.1	6.4	-
	6	8.0	6.5	5.7
Concora crush (N)	2	-	(270)	-
	4	370	335	-
	6	380	365	310

* Source: Farrington, Hansen and Nelson (1976): Utilisation of Young Plantation *E. globulus*.

TABLE 8.7

Influence of some arithmetic ratios on the pulp strengths (Kraft) of fertilised and unfertilised 6 year old *E. globulus* debarked wood.

Treatment (fertiliser)	FL (mm)	D u	W u	L u	FL/D (felting co-efficient)	L/D	2 W/L (Runkel)	Burst Index	Tear Index	Breaking Length (km)
A (0.0 kg/ha)	0.95	15.7	2.8	10.1	61	0.65	0.55	8.5	10.1	12.2
B (565 kg/ha)	0.90	15.8	2.5	10.8	57	0.68	0.46	8.6	10.3	12.6
D (1130 kg/ha)	0.87	17.0	2.5	12.0	51	0.71	0.42	8.7	10.8	12.1

FL = Fibre length

D = Fibre diameter

W = Fibre wall thickness

L = Fibre lumen diameter

TABLE 8.8

Summary of statistics of linear regression Y = a+bX of some anatomical wood characteristics and beaten pulp properties of *E. globulus*. (Y = burst, tear index and breaking length and X = anatomical characteristics as shown in the Table).

PROPERTY	NUMBER OF OBSERVATIONS	a	b	CORRELATION COEFFICIENTS r	r^2
Burst x Runkel ratio	3	9.276	-1.419	-0.997*	0.993
" x FL/D ratio	3	9.854	-0.022	-0.998*	0.996
" x Basic density	3	11.233	-5.187	-0.993	0.985
" x Fibre length	3	9.729	-1.283	-0.934	0.873
" x Lumen diameter	3	7.495	0.100	0.986	0.972
" x Fibre diameter	3	6.344	0.140	0.918	0.842
" x L/D ratio	3	6.661	2.838	0.997*	0.993
" x Double wall thickness	3	10.036	-0.278	-0.914	0.836
Breaking length x Double wall thickness	3	13.234	-0.181	-0.225	0.051
" x Runkel ratio	3	12.107	0.405	0.108	0.012
" x FL/D ratio	3	11.464	0.015	0.252	0.063
" x L/D ratio	3	13.685	-2.027	-0.269	0.072
" x Basic density	3	11.815	0.955	0.069	0.004
" x Fibre diameter	3	15.966	-0.228	-0.564	0.318
" x Lumen diamater	3	13.342	-0.950	-0.351	0.124
" x Fibre length	3	19.098	-7.239	-0.999*	0.998
Tearing index x Fibre length	3	17.553	-7.898	-0.921	0.973
" x Fibre diameter	3	1.658	0.544	0.986	0.973
" x L/D ratio	3	3.474	10.135	0.987	0.975
" x Basic density	3	19.338	-17.607	-0.935	0.874
" x Lumen diameter	3	6.371	0.367	0.997*	0.995
" x Double wall thickness	3	14.877	-0.869	-0.790	0.625
" x FL/D ratio	3	14.859	-0.079	-0.984	0.968
" x Runkel ratio	3	13.040	-5.608	-0.975	0.949

* P = 0.05
$r_{0.05}$ = 0.997

FL = Fibre length

L = Fibre lumen diameter

D = Fibre diameter

Runkel ratio = Double wall thickness/Fibre lumen diameter

TABLE 8.9

Relation of some anatomical properties to some Eucalypt paper strength properties

(Sulphate pulps)*

SPECIES	FL (mm)	D u	W u	L u	FL/D (felting co-efficient	L/D	2W/L (Runkel)	Breaking Length (km)	Burst Factor	Tear Factor
E. regnans F. Muell	0.70	19	3.1	12.8	40	0.67	0.48	10.7	71	81
E. deglupta Blume	0.90	20	2.7	14.8	44	0.73	0.37	8.7	57	104
E. moluccana Roxb†	0.90	20	6.6	6.8	47	0.34	1.94	3.1	12	32

FL = Fibre length

D = Fibre diameter

W = Fibre wall thickness

L = Fibre lumen diameter

* Source: Watson and Dadswell (1964)

† Formerly *E. hemiphloia* F. Muell ex Benth (Johnson and Maryatt, 1965)

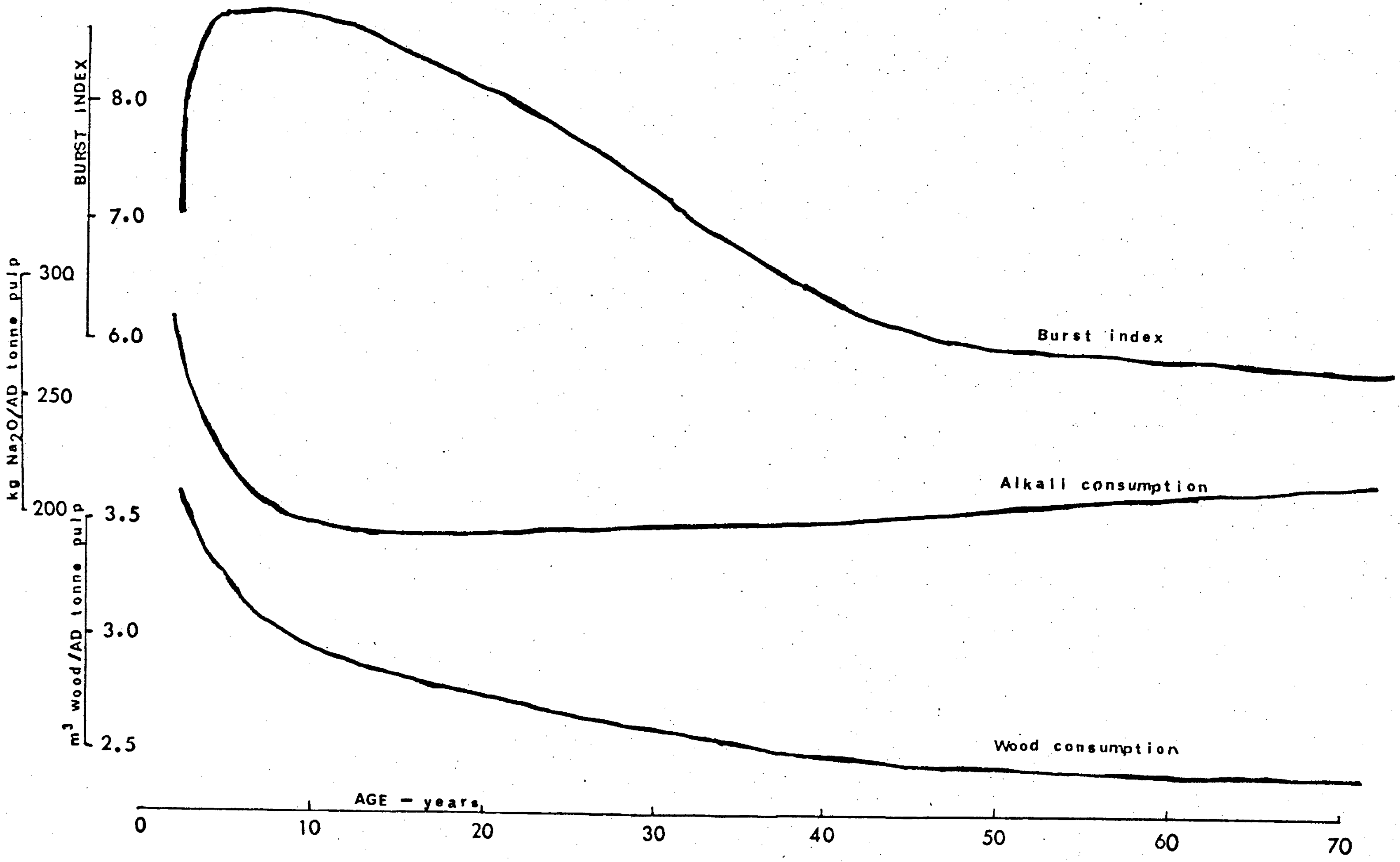

Fig. 8.1. The variation of important pulping characteristics of *E. globulus* regrowths with age.

CHAPTER 9

SUMMARY AND CONCLUSIONS

9.1 Summary

The effect of fertilisation on the anatomical and chemical characteristics of *E. globulus* and their influence on the properties of pulp and paper derived from this species were studied. The wood samples were taken from 6-year old *E. globulus* trees, grown in a replicated fertiliser experiment. These had been established in 1969 at the A.P.M. Forests Pty. Limited, Silver Creek Farm, south-east of Morwell, victoria. The experiment involved four levels of fertiliser addition with four replications. The fertiliser was a blend of ammonium phosphate and ammonium sulphate (HyGold 18) containing 18% nitrogen and 8% phosphorus. It was applied by hand to the surface of the soil over a period of 15 months.

Four trees were randomly selected from each treatment (A, B, C and D with 0.0, 188, 565 and 1130 kg/ha of fertiliser respectively). One tree was selected for height sampling from Treatment C, the selected tree being the one closest to the mean diameter of Treatment C plots. The trees were felled and discs were taken at breast height from the sample trees of the treatment plots and from the selected tree for height sampling. Discs were taken at five different sampling heights (0.25, 1, 3, 5 and 7 metres above ground level). Radial strips were taken from each disc and these were used for the fibre length, double wall thickness, lumen diameter and percentage volumes of the cell types.

For the fibre length determination the strips were divided into different growth rings (1969-1974) macerated, and the fibre suspension stained before mounting. Using a projecting microscope, a total of 50 whole fibres were measured for each growth ring to obtain the average fibre length.

For determining the double wall thickness, lumen diameters and the percentage volume of cell types 1 cm cubes were taken from the radial strips. One hundred measurements were made on each of the cubes; 50 for double wall thicknesses and 50 for lumen diameters. Ten transects were made on the cross-section of each cube and one on the tangential section to determine volume of rays. The tangential volume percent of rays was used to adjust the differences between the measurement of the two surfaces to obtain the average percentage volumes of cell types.

The basic densities were determined by direct measurement of the oven-dry weight and the determination of the swollen volume by the Heinrichs and Lassen (1970) method. The amount of ether- and methanol-soluble extractives in the wood and bark samples from each disc were determined on ground dried samples. The percentages of the amount of extractives (based on o.d. wt of wood) ranged from 1.32-1.56 (ether-soluble), 2.03-4.38 (methanol-soluble) for wood; 2.20-2.48 (ether-soluble) and 9.02-9.79 (methanol-soluble) for bark.

Pulp and paper properties of the wood from treatment D, the highest level of fertilisation, were determined. The aspects studied included pulping processes, cooking conditions, amount of chemicals and beating on pulp properties of debarked stemwood. The processes used were NSSC "C" (soda type), pure soda and Kraft. The data on Kraft pulp properties of three treatments (A, C and D) and Kraft and NSSC pulp properties of debarked stemwood, unbarked stemwood and branches with leaves of Group D trees were provided by the A.P.M. Pty. Limited (Farrington, Hansen and Nelson, 1976).

The trees were randomly selected from Treatment D, one group approximating the mean diameter of the treatment plots and two groups with diameter about 1.6 standard deviations above and below the mean trees. In all, 24 trees were taken from treatment. The sample preparation involved felling, chipping to correct size and charges equivalent to 400g of oven-dry chips were cooked by the different processes using the same liquid to wood ratio of 3.5 ml/1 gm oven dry wood. The cooking conditions and amounts of cooking chemicals are shown in Table 8.3. The same sampling and preparation procedures were also used in all other investigations for which data were supplied. The anatomical properties were regressed against the resultant properties of the pulp and paper derived from the wood.

The average basic densities were not significantly different between the 4 treatments. However there was a decrease in basic density from the lowest level of fertilisation (Control, 0.0 kg/ha) to the highest (Group D, 1130 kg/ha), although still only a 7% decrease from Control A (Table 3.4). The basic density increased with increasing height.

The average fibre lengths of the first and second growth rings of the Control group were significantly higher than those of Group D at the 5% level of probability (Table 4.1(a)). The average fibre lengths for the remaining four growth rings were not significantly different for all treatments. In the comparison of the average fibre lengths of the different treatments no significant difference was detected. Thus fibre length is only significantly reduced in the early years by higher level of fertilisation application. There is a small decrease in the average fibre length as the level of fertilisation increases, the biggest decrease of 9% occurring in Group D, compared wiht Control (Table 4.1(b)).

The lumen diameters at the higher levels of fertilisation were significantly greater than those of the Control and Group B. The double wall thickness is only significantly affected at the highest level of fertilisation although all the fertilised groups had lower double wall thickness and greater lumen diameters (Table 5.1 (b)). There was no significant difference in both parameters for all the sampling heights.

The volume of tissue by percentages was not significantly different for the four treatments although there was a progressive increase in the proportion of volume of fibres as the level of fertilisation increased. Lower parenchyma volumes were closely associated with larger fibre volumes. In the regression analysis of double wall thickness, lumen diameter and fibre diameter against basic density and fibre volume, double wall thickness and lumen diameter were significantly correlated negatively with fibre volume and basic density respectively at the 5% level of probability. The fibre volume increased with increasing height though not significantly.

The amount of ether-soluble extractives in the wood from the four treatments was not significantly different and was the same in the bark samples. The methanol-soluble extractives content of the wood at the highest level of fertilisation was significantly higher than that of the other three groups. The amounts in the bark of all groups were not significantly different although fertilised groups had progressively higher amounts of both ether- and methanol-soluble extractives (Tables 7.2 (a) and (b)).

In the tree selected for height sampling the total amounts of methanol- and ether-soluble extractives in the wood at the different sampling heights were not significantly different. In the bark, however, the three last sampling points (3, 5 and 7 m) had significantly higher amounts of total methanol- and ether-soluble extractives compared with points 0.25 and 1 m. While the variation of total amounts of extractives with height for bark was that of increase with increasing height, that for wood was not a clear trend (Table 7.1 (b)).

The lignin and pentosan contents of the wood at the highest level of fertilisation (Group D) were lower than those of the Control but not significantly so, while the boiling water extractives were higher than the Control but not significantly so.

The fertilised groups produced more volume increment per year than the Control. Group C at age 2 years produced almost ten times more dry weight of wood than Control, but at age 6 years Group D, the highest fertilised group, was producing four times more wood than Control. Thus there is a levelling off of effect of fertilisation in late years.

The individual tree response was not measured in this study, as average values of all the determined characteristics were used in the analysis of results. However, a closer look at the data for individual trees indicated some individual responses. Tree 39 from the Control produced less dense wood than Tree 41 fertilised at the 188 kg/ha rate but a higher fibre length. Tree 21 from Group C produced a denser wood and higher initial fibre length than Tree 18 from Group B. Individual trees need further investigation since this approach could provide a basis for selecting individual trees for desirable end product characteristics.

In the pulping studies using different processes, the NSSC "C" soda type cook, produced higher yields when compared with the soda and Kraft cooks. However, the Kraft pulp strengths were higher initially and responded to beating better, by rapid increases as compared to other cooks (Tables 8.4(a),(b) and (c)). Also Kraft pulps had lower kappa number and the increase in the amount of chemicals in all cooks was followed by a lowering of kappa numbers. The pulps of the NSSC "C" soda type cook were very low in brightness compared with Kraft and soda cooks.

The data on the Kraft pulp properties of the woods from 3 fertiliser treatments showed that the Group D trees produced pulps of higher burst and tear strengths but lower breaking length as compared with Control pulps. Correlating these data with the anatomical characteristics, one immediately sees that Group D trees have lower basic densities, relatively thinner walls and wider lumen diameters. This reflects the importance of the amount of collapsibility of fibres and the accompanying increased inter-fibre bonding resulting in a more compact sheet with higher strengths.

In the regression analysis of some of the anatomical characteristics and the strength properties of Kraft pulps from the three fertiliser treatments, Runkel ratio, felting coefficient (fibre length to fibre diameter) and the ratio of lumen diameter to fibre diameter correlated significantly with burst index, the first two negatively, the latter positively. Fibre length correlated negatively and significantly with breaking length. Lumen diameter correlated with tear index significantly and positively (Table 8.8).

The importance of longer fibre length in developing tear strength did not show up in this study. (The correlation of fibre length with tear index was poor.) The fibre length data range was small and this could have contributed in masking the influence of fibre length.

The inclusion of bark and branches to the wood of Group D samples resulted in an increase in yields of pulp per hectare per year from both cooking processes. There was, however, a significant reduction in the pulp strengths for

NSSC corrugating grades. The reductions in Kraft and NSSC strong grade pulps were small. However, inclusion of these portions of the trees increased chemical consumption significantly both in cooking and bleaching. This was due to the higher extractives content, especially the methanol-soluble fractions. The potential burning properties of the black liquor, on which effective recovery procedures are based, was significantly decreased due to the inclusion of the same fractions.

Variations in wood characteristics between trees occur partly because of genetic differences, partly as a response to the different growth conditions. Fertilisation can affect both genotypic and phenotypic variation, the extent of which was not measured in this study, so that the results are mainly mean values which show trends only. The experiments should, therefore, be viewed in the light of these limitations to avoid incorrect generalisations. It should also be emphasized that many investigations on the effects of fertilisation have not been carried out long enough to yield long term results and in other cases other important parameters have not been taken into account sufficiently. Most past studies were based on effects before and after fertilisation and a number of other influences, such as age, climate and stand density could distort results.

9.2 Conclusions

A high degree of fertilisation produces wood of a lower basic density than the Control, the decrease is not, however, significant. The significant increase in volume of wood produced should more than compensate.

High levels of fertilisation significantly reduce double wall thickness and increase lumen diameter. This increases the amount of relatively thin-walled fibres allied with only a slight non-significant reduction in fibre length; this makes them more suitable for pulp and paper since the fibres collapse more readily and give higher strengths in the pulp and paper produced from them.

Fertilisation significantly increases the extractives content of wood but only at the highest level of addition. Though this will lead to higher chemical consumption during pulping, it will only be of significance when bark and branches are also added to the wood.

The inclusion of bark does not significantly affect the strength of pulps except in the NSSC corrugating grades.

It is recommended that an optimum utilisation age of 10 years for fertilised *E. globulus* be considered. However this needs to take into consideration the cost element in wood procurement. As found in some of the properties investigated, there is a levelling off of effect of fertilisation as the trees grow older, consequently it is suggested that the application of fertiliser be spread over a few years.

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